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# VON KARMAN CENTER

SNAP-8 DIVISION

## SNAP-8 MATERIALS REPORT FOR JANUARY-JUNE 1965

BY H. DEROW AND B. E. FARWELL

PREPARED FOR

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

CONTRACT NAS 5-417

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SEMIANNUAL REPORT

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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

July 1965

CONTRACT NAS 5-417

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# FOREWORD

Aerojet-General Corporation is proceeding with the design and development of the SNAP-8 Power Conversion System, as authorized by National Aeronautics and Space Administration Contract No. NAS 5-417.

The ultimate objective of the SNAP-8 Program is to design and develop a 35-kw electrical generating system for use in various space missions. The power source will be a nuclear reactor furnished by the Atomic Energy Commission. The SNAP-8 system will use a eutectic mixture of sodium and potassium (NaK) as the reactor coolant and will operate on a Rankine cycle, with mercury as the working fluid for the turbogenerator. The SNAP-8 system will be launched from a ground base and will be capable of unattended full-power operation for a minimum of 10,000 hours. After the system is placed into orbit, activation and shutdown may be accomplished by ground command.

This semiannual materials report is submitted in partial fulfillment of the contract and covers the period from 1 January through 30 June 1965. Part of the information was prepared at Aerojet-General Nucleonics, San Ramon, California, under Aerojet-General Corporation Subcontract 274949.

The Component Materials Development Program was under the management of R. S. Carey, Head, Technical Support Department, SNAP-8 Division, Von Karman Center. The work at the Von Karman Center was done under the direction of H. Derow, Head, Materials Section, SNAP-8 Division. The work at Aerojet-General Nucleonics was done under the direction of B. E. Farwell, Head, SNAP-8 Section, Metallurgy Department, Applied Science Division. The following engineers contributed to the various programs:

At Von Karman Center - H. Bleil, F. Cassidy, and R. McPherson

At Aerojet-General Nucleonics - A. R. Herdt, G. Redfern, E. F. McDaniel, J. H. Ralphs, and M. K. Wong.

The suggestions and guidance of W. Titus and M. Parkman, Metallurgy Department, Aerojet-General Nucleonics, and of P. Stone, SNAP-8 Project Office, Lewis Research Center, NASA, are gratefully acknowledged.

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Report No. 3038

NASA CONTRACTOR REPORT

# SNAP-8 MATERIALS REPORT FOR JANUARY-JUNE 1965

Aerojet-General Corporation

## ABSTRACT

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Investigations were conducted in support of the design, fabrication, and development testing of various SNAP-8 components.

Investigation of a mercury forced-convection corrosion loop was continued with the aim of evaluating the corrosion resistance of 9Cr-1Mo alloy steel (the reference mercury-containment material) for 10,000-hour service. Rubidium is being evaluated as an additive to the mercury to promote boiler conditioning.

Data were developed in a continued program to evaluate the effect of the SNAP-8 operating environment on 9Cr-1Mo steel.

author

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## GLOSSARY

Abbreviations commonly used in the SNAP-8 Program are defined below.

| | |
|---|---|
| AA | Alternator assembly |
| AEC | Atomic Energy Commission |
| AGC | Aerojet-General Corporation |
| AGN | Aerojet-General Nucleonics |
| AI | Atomics International |
| AZFO | NASA-Azusa Field Office |
| CL | Corrosion loop (AGN) |
| CTL | Component-test loop (AGN) |
| DDAS | Digital data acquisition system |
| DWG | Drawing |
| EDM | Electrical-discharge machining |
| EFF | Efficiency |
| EGS | Electrical generating system |
| EM | Electromagnetic |
| EME | Electromagnetic equivalent |
| FPS | Flight-prototype system |
| FPTF | Flight-prototype test facility |
| GE | General Electric Company |
| GPS | Ground-prototype system |
| GPTF | Ground-prototype test facility |
| HR | Heat rejection |
| HRF | Heat-rejection fluid |
| HRL | Heat-rejection loop |
| HRS | Heat-rejection system |
| HTL | Heat-transfer loop |
| L/C | Lubricant/coolant |
| LeRC | Lewis Research Center |
| LML | Liquid-mercury loop |
| LNL | Liquid-NaK loop |
| LPL | Low-power loop |
| MIS | Mercury-injection system |
| Mix-4P3E | Bis(mix-phenoxyphenyl) ether, a mixture of the six possible isomers of bis(phenoxyphenyl) ether, considered as a lubricant/coolant for the PCS |
| ML | Pyre-ML, Du Pont polyimide organic resin |
| MPMA | Mercury pump-motor assembly |
| NaK | Eutectic mixture of sodium and potassium |
| NASA | National Aeronautics and Space Administration |
| NHRA | NaK heat-rejection assembly |
| NPA | NaK pump assembly |
| NPMA | NaK pump-motor assembly |
| NPS | Nuclear power system |
| NPSH | Net positive suction head |
| NS | Nuclear system |
| NSL | NaK-simulation loop |
| ORNL | Oak Ridge National Laboratory |
| PCS-1 | Power Conversion System 1 |
| PF | Power factor |
| PL | Primary loop |
| PLR | Parasitic load resistor |
| PMA | Pump-motor assembly |
| PNLA | Primary NaK loop assembly |
| PO | Purchase order |
| PVT | Pressure-volume-temperature |
| $R_B$ | Rockwell B (hardness) |

## GLOSSARY (cont.)

| | |
|---|---|
| RPL | Rated-power loop |
| SC | Speed control |
| S8DS | SNAP-8 development system |
| S8ER | SNAP-8 experimental reactor |
| SL-1 | System Loop Test Facility 1 |
| SNAP | Systems for Nuclear Auxiliary Power |
| SR | Saturable reactor |
| SS | Stainless steel |
| TA | Turbine assembly |
| TAA | Turbine-alternator assembly |
| TCL | Thermal-convection loop (AGN) |
| TR | Transformer-reactor (assembly) |
| TRW | Thompson Ramo Wooldridge |
| TS | Test section |
| TSE | Test-support equipment |
| T-T | Tube-in-tube |
| VR | Voltage regulator-exciter |
| WOO | Western Operations Office |
| -X<br>-1<br>-2<br>-3 | Standing alone (i.e., not preceded by letters of the alphabet), these designations indicate design stages of SNAP-8 hardware |

## SYMBOLS*

| | |
|---|---|
| P | Pressure, psia ($P_H$ system = high-pressure system, $P_L$ system = low-pressure system) |
| T,t | Temperature, $^oF$ |
| TIR | Total indicator reading |
| $\dot{W}$ | Flow rate, lb/hour |
| $\Delta$ | Difference |

Subscripts

| | |
|---|---|
| b | Boiler |
| H,Hg | Mercury |
| i,in | Inlet |
| N | NaK |
| o,out | Outlet |

*Used in illustrations and not defined there.

# SNAP-8 MATERIALS REPORT FOR JANUARY-JUNE 1965

by H. Derow and B. E. Farwell

AEROJET-GENERAL CORPORATION

## SUMMARY

Work was performed during the first half of the 1965 calendar year with the aim of providing data to guide the selection of materials for SNAP-8 system components and of providing metallurgical assistance in the design, development, fabrication, and testing of that system. This work is summarized below under the applicable task categories.

### Component Design and Development Support

A high starting torque exhibited by the NaK (eutectic mixture of sodium and potassium) pump-motor assembly (PMA) during water testing was attributed to a high coefficient of static friction of the NaK-lubricated journal bearing. Static friction tests were conducted to evaluate the possible effect of various bearing-surface conditions. Molybdenum disulfide most effectively reduced the coefficient of friction (to 0.09); however, it is nonpermanent in a unit subjected to many starts and stops. Electrolized chromium, with a static coefficient of friction of approximately 0.16, appears to be an acceptable compromise between molybdenum disulfide and uncoated surfaces (coefficient of friction, 0.40).

### Component Fabrication Support

A production order was completed for 30-ft-long tubes of 9Cr-1Mo steel (alloy including 9% chromium and 1% molybdenum) required for tube-in-tube (T-T) boiler fabrication. Twenty-three of 27 tubes rejected by ultrasonic inspection were made usable by grinding out surface defects that were 0.003 to 0.020 in. deep (predominantly 0.006 in.). Metallographic examination indicated that the defects were probably caused by excessive inclusions in the starting raw material. A cleaning procedure was devised for removing a tightly adherent oxide film produced during the final heat treatment of these tubes in an exothermic gas atmosphere.

Experiments indicated that tree-resin reinforcement is required in the annulus formed between the mercury-containment tubes and the outer 321 SS (stainless steel) tube when coiling the T-T boiler. The internal area of the 9Cr-1Mo mercury-containment tubes requires no reinforcement when a 34.5-in.-dia boiler-coil assembly is bent, but tree resin is required for a 20.5-in.-dia assembly. Double-cycle cleaning with an alkaline solution (MIL-C-14460, Type I) will remove the tree resin after coiling.

Test Operations Support

Before Rated Power Loop 2 (RPL-2) was operated in February 1965, the mercury-containment tubes of the boiler were cleaned to remove possible contamination generated during the previous operating period (rubidium was added to the mercury to promote satisfactory boiler performance). Cleaning Method II in Aerojet Specification AGC-10319/6 was used. A test run in February 1965, lasting approximately 100 hours, did not include rubidium and the boiler never achieved the rated mercury-output conditions. A test run in March lasting more than 100 hours included a single rubidium injection, which was sufficient to maintain satisfactory boiler performance throughout the run. Although some rubidium was apparently lost during three shutdowns, the amount remaining was sufficient for whatever boiler surface conditioning was necessary. A test in May-June 1965 was conducted with no rubidium additive, and it was found that none was required for satisfactory boiler performance. It appears that (1) the rubidium first served as a "conditioning agent" during what would otherwise have been a nonperforming run-in period, and (2) as the operating duration increased, the boiler was able to perform properly without the additive.

During this last operating period, periodic residue and mercury samples were taken from the loop after shutdowns. The mercury remained free of rubidium. The residues contained rubidium, probably as oxides formed during previous operating periods and retained in the loop. Rubidium was detected in deposits on piping or component surfaces, or in residues presumably washed into the dump tank during mercury dumps. Rubidium has been continually detected, in ever-decreasing amounts, in all residue samples analyzed during this report period. These analyses also indicated that organic materials were entering the loop. Mix-4P3E* was detected, as were various oils used in vacuum pumps in the system. The specific source of these fluids is not presently apparent.

A particle was found lodged in one tube-inlet-restrictor orifice of the RPL-2 boiler; potential sources in the loop are the turbine-simulator lines and some parts of the boiler. Solid particulate material was found clogging the jet-pump orifice of the mercury PMA. Chemical analysis and metallographic examination indicated a mixture of metallic, chip-like particles and weld spatter. One nonmagnetic particle, analyzed as Stellite 6B, was judged to have been a part of the trailing edge of a turbine nozzle. A wet brown residue was found in the standoff line of an RPL-2 turbine-pressure transducer; it had not affected the function and calibration of the transducer.

The throat section of the NaK-flow venturi in the NaK primary loop of RPL-2 was examined after approximately 800 hours of operation, most of which had been accumulated without oxygen control in the loop. The inlet and throat contained relatively heavy deposits; the outlet contained only a small amount. Water-flow tests indicated that the venturi readings would imply flow rates 25% higher than the actual rate. A sample of the residue, analyzed qualitatively by emission spectroscopy, was found to contain chromium, nickel, and molybdenum. Sections of the RPL-2 loop piping at the inlet and outlet of the gas-fired NaK

*See Glossary.

heater were examined. The microstructure of the inlet section indicated a surface deposit and also an intergranular deposit extending approximately 0.001 in. (cross sectionally) into the pipe. The microstructure of the outlet piping indicated the possible presence of minor deposits, but there was no evidence of intergranular penetration. Mass-transfer deposits were found in the pumping section of the RPL-2, primary, NaK, electromagnetic (EM) pump; they caused reduced NaK flow through the pump at the rated current flow. The deposits were hard and tenacious and appeared to be concentrated in bus-bar-attachment areas.

An analysis was performed on the RPL-2, 316 SS, NaK-to-NaK, heat exchanger that failed at the outlet end after 1356 hours and 47 cycles of primary-loop (PL) operation and 1170 hours and 44 cycles of heat-rejection-loop (HRL) operation. Operational stresses imposed on the heat exchanger at the weld connecting it to the heavy-walled loop piping apparently exceeded the ultimate strength of the thin exchanger-shell material, resulting in component failure. Evidence was found that both metal mass transfer and carbon transport had occurred.

A graphite-base heat-transfer cement (Thermon Type T-63) was applied to provide a heat-transfer medium between trace heaters and the liquid-metal lines of RPL-2. Examination during a loop shutdown in April indicated that cement degradation had occurred on several lines. In one instance, the cement temperature was below the maximum use temperature specified by the vendor. In another instance, improper control of NaK-line heaters raised the cement temperature significantly above the specified maximum of 1250°F. The degradation resulted from oxidation of the graphite and melting of the cement binder adjacent to the heater sheaths. The heat-transfer cement was thus converted to an insulating material. No evidence of cement degradation was apparent on the lines maintained at 600°F or lower.

Precipitated crystals, primarily p-p isomer, were found in a sample from the first production order of mix-4P3E for the lubricant/coolant (L/C) loop of RPL-2. The cause of the precipitation appeared to be a p-p isomer content of the fluid above a critical amount for the maintenance of chemical stability (3%). A sample of the mix-4P3E from RPL-2 was analyzed after the turbine-test series. Approximately 800 hours of testing had produced very little physical-property change. Fluid discoloration appears to have been caused by partial thermal decomposition above 800°F. Overheating of the fluid at the Calrod heater surfaces was evidenced by a carbonaceous deposit on the heater sheath. At Aerojet's request, Shell Development Company ran dynamic viscosity tests on mix-4P3E, using a Hoake Rotovisco (rotary viscometer). Tests at 68, 122, and 176°F indicated that the fluid is Newtonian in its behavior.

A pipe failure occurred in the HRL purification system of RPL-2. The failure appeared to be associated with a trace heater used to maintain the pipe temperature during loop operation. Five holes penetrated through the pipe wall within an 8-in. length in the failure area. The microstructure indicated that the 316 SS had been molten in the area of each hole. A probable cause of the failure was electrical shorting of the heater through the pipe to ground.

Power Conversion System 1 (PCS-1) of System Loop Test Facility 1 (SL-1) was cleaned prior to final assembly. The cleaning procedure employs an alkaline rust remover (including approximately 70% sodium hydroxide) and an alkaline detergent. The turbine-alternator assembly (TAA) and mercury PMA were not cleaned in this fashion, but individual parts were ultrasonically cleaned in Freon during assembly. All 9Cr-1Mo, PCS-1, mercury-loop tubing was X-rayed because metallographic examination of samples revealed internal-surface lap-type defects. The defective material was replaced.

Sections of three trace heaters removed from the L/C line of SL-1 were evaluated after two of them had failed. Metallographic and radiographic examination indicated that the failure occurred because the Nichrome heating wire became overheated, melted, and flowed through cracks in the magnesium oxide insulation to short the heating wire to the sheath. It is believed that the cracks were produced when the heaters were bent to fit the contour of the loop pipe. The primary cause of the overheating of the heater apparently was the unintentional presence of thermal-insulation material between the heater and the pipe in localized areas.

Residue samples from the NaK HRL Chempump that failed after a few seconds of rotation during a wet shakedown test of SL-1 were collected and analyzed. They appeared to consist of a mixture of finely divided oxides of NaK, metallic particles resulting from pump-bearing galling during the failure, abrasive residue that was not completely removed from a pump component after a powder blasting or grinding operation, and a polymer material.

Two NaK PMAs and Liquid-NaK Loop 3 (LNL-3) were flushed with alcohol and water to remove oxide buildup caused by loop operation without a NaK-purification system. When the last pump was cleaned by internal flushing, a black residue was found in the cleaning fluids. Infrared spectrophotometry produced a representative curve that was not identifiable. Two NaK PMA housings were evaluated after NaK leaks developed during LNL-3 tests. It was found that the castings contained excessive porosity and, in one instance, a casting cold-shut resulted in an unfused through-the-wall plug. Through-wall porosity in unused housing castings was not detected in dye-penetrant tests or single-wall radiography. A housing that had failed due to through-wall leaks was brush-plated with iron and was returned to testing; through-wall leakage again occurred during a subsequent test run.

A paste-like Cerrobend residue was found in the visco pump seal of a mercury PMA tested in Liquid-Mercury Loop 3 (LML-3). A black, flaky residue was also found in the mix-4P3E fluid of the same loop system. The LML-3 was apparently assembled in a manner that did not assure overall system cleanliness.

### Transformer-Reactor Assembly, Heat-Sink Transition Joint

Electroless nickel, silver over electroless nickel, and Electrolized chromium were evaluated as candidate coatings to protect the copper portion of the transition joint against reaction with the L/C fluid (mix-4P3E) in a nuclear-radiation environment. A galvanic-corrosion reaction was found to have occurred between the aluminum portion of the transition joint and the nickel during the plating of the specimen. The Electrolized chromium coating exhibited satisfactory adherence, but coating voids were found on the unexposed specimen and coating cracks were found on the exposed specimens. An evaluation of experimental, direct-bond, aluminum/316 SS specimens that were exposed to mix-4P3E at 250°F was also completed. The specimens appeared unaffected. Metallographic examination showed no surface reaction with the fluid, nor was there evidence of cross-bond diffusion resulting from the elevated-temperature exposure.

## Bimetal-Tube Evaluation

A 9Cr-1Mo/316 SS tube butt-weld joint specimen was prepared by TIG (tungsten, inert-gas) welding. Metallographic examination of sample sections indicates the retention of individual layers of 9Cr-1Mo steel and 316 SS in the welded area. The 9Cr-1Mo steel liner collapsed, however, probably because it was too thin to support the thermal stresses.

Twelve tube lengths of two different sizes of columbium/316 SS tubing were manufactured by Nuclear Metals, Inc., Concord, Massachusetts (0.400- and 0.684-in. inside diameter (ID) by 0.055-in. wall, of which 0.020 in. was columbium). Ultrasonic inspection and confirming metallographic examination indicated that only three lengths (none of the large-ID specimens) contained an acceptable metallurgical bond ($<$5% unbond) between the columbium and the 316 SS.

## Boiler Conditioning Studies

An investigation of the boiler-performance effect of adding rubidium to the mercury was initiated. Apparatus was designed and fabricated for making controlled rubidium additions or a mix-4P3E addition to the mercury in Component Test Loop 2 (CTL-2) at Aerojet-General Nucleonics (AGN). The loop was started and was brought to rated boiler-outlet conditions (265 psia at 1265°F). Twenty-five grams of mix-4P3E was admitted to the mercury-pump suction. An effect on boiler conditions was noted immediately, and within 45 min the boiler-outlet temperature had dropped from 1270°F to 1100°F, indicating that the boiler was deconditioned. Small amounts of rubidium were subsequently added to the mercury in the loop through an auxiliary addition tank. Partial conditioning (boiler-outlet condition, 1270°F and 165 psig) was indicated after the rubidium concentration reached 600 ppm. This partial conditioning of the boiler could not be sustained when the boiler-outlet pressure was increased, as indicated by a decrease in the boiler-outlet temperature. The rubidium content of the mercury appears to decay if constant additions are not made.

## 9Cr-1Mo Steel, Strength Evaluation

Eight weld specimens were tested in air at 1325°F for up to 1845 hours. The tests indicate that welding does not decrease the creep or creep-rupture strength of 9Cr-1Mo steel at 1325°F. Cyclic-creep tests were completed on 9Cr-1Mo steel capsule specimens in a NaK environment at 1325°F. The internal pressure was changed each 30 sec so that a $\pm$10% stress cycle resulted.

Analysis of capsule-wall measurements revealed that creep occurred in all sections. Micrographic examination showed that severe grain growth occurred and was accompanied by elimination of the carbide precipitation in the grain boundaries. A fine dispersion, appearing to be carbides, remains equally distributed throughout the wall, except for a layer less than 1 mil thick on the inside surface. The same precipitation pattern was noted in the unmachined sections of the walls, but some parts of these lower-stressed sections did not recrystallize.

## Corrosion-Loop Program

Corrosion Loop 3 (CL-3) was removed from its test cells and disassembled after a section of 316 SS tubing failed in the NaK heater, causing a shutdown. The failure resulted from intergranular cracking associated with a second phase that precipitated heavily at the grain boundaries, especially in the failure area. Selective etching indicated the precipitate may be sigma phase.

The boiler performance constantly improved during the first 2000 of a total 4400 hours of operating time. The 316 SS outer shell of the mercury boiler showed no evidence of corrosion. There was some carburization of the 316 SS in the hottest section of the boiler (1310°F). The NaK side of the 9Cr-1Mo mercury-containment tubing exhibited decarburization, primarily at and near the NaK-inlet end. The decarburization depth decreased gradually along the boiler toward the NaK outlet, where the temperature had decreased to approximately 1270°F. No decarburization was observed in tube sections where the NaK temperature was below 1270°F. Some exterior cracking of the 9Cr-1Mo steel tubing was also observed, most of it in the area of highest heat transfer. Tube cracking was also observed on the mercury side (interior). Pitting was found on the 9Cr-1Mo steel boiler-inlet plug (5 ft long) in the area 7 to 21 in. from the boiler inlet. The inner surface of the 9Cr-1Mo steel tubing in the plug area was lightly pitted. The NaK-temperature profile of the boiler indicates practically no heat transfer in the plug region after the preheating of the liquid mercury in the first 2 ft of the boiler. Examination of the interior of the 9Cr-1Mo steel boiler tubing following the plug region indicated heavy pitting and surface cracking in some sections, especially where the heat transfer from the NaK to the mercury was the greatest. Corrosion-product deposition was also found in that area.

Other NaK-primary-loop components (e.g., the EM pump, EM flowmeter, and 316 SS tubing) showed no indications of corrosion or mass transfer. Sections of Chromalized Hastelloy N, 347 SS, and Hastelloy C incorporated in the NaK primary system to simulate reactor materials showed no appreciable corrosion. The 347 SS in a high-temperature section of the NaK primary system (1320°F) showed less change in microstructure than the 316 SS.

Most of the corrosion-product deposition in the mercury loop was found 25 to 30 ft from the boiler inlet, where the pitting depth increases sharply. The corrosion products are deposited from supersaturated liquid mercury in the liquid vapor stream as the vapor quality approaches 100%.

The change in the NaK-temperature profile with operating time indicates that the mercury-flow pattern was changing constantly up to 2000 hours of operation. This would account for the pitting and corrosion-product deposition observed in the region 30 to 50 ft from the mercury inlet.

Other areas of the mercury system appear to be free of serious corrosion and mass-transfer problems when 9Cr-1Mo steel is used. Essentially no mass-transfer deposits were found in the condenser and the liquid lines in the loop.

The corrosion products generated in these areas apparently remained suspended in the mercury and/or floated at the mercury interfaces.

Examination of the NaK side of the mercury condenser, the NaK heat exchanger, and the tubing that made up the condensing system revealed no mass transfer or corrosion. Because of the low operating temperature of this system (700°F maximum), no material-corrosion problem was expected.

CL-4 was operated for 1100 hours. Oxide control of the NaK primary loop was satisfactorily maintained by cold trapping. Superheated vapor was obtained immediately when mercury was injected into the boiler, but rated design conditions were not obtained.

A modified mercury-inlet-plug insert was installed in the boiler to replace the original plug. The geometry of the preheating region of the modified plug insert was changed to increase the liquid-mercury flow rate from 0.91 to 6.22 fps. A test run with the modified plug showed an immediate heat-transfer improvement in the inlet-plug region and completion of mercury boiling at a length of 30 ft. Considerable conditioning was noted during the subsequent 16 hours of operation. The boiler was then shut down and restarted to confirm the conditioning phenomena. Upon restarting, it exhibited the same heat transfer and stable boiling performance as before.

A boiler-inlet-plug test program was initiated. Four different instrumented plugs were tested for design evaluation, and a run was also made without a plug insert. The corrosion patterns produced in these tests indicate that the increased liquid velocity and improved boiler performance caused noticeable corrosion in plug regions with operating times as short as 100 hours. It was also found that the clearance between the inner surface of the boiler tubing and the outside of the plug was critical. If this clearance is not controlled, the differential pressure and liquid velocity in the tight-pitch region of the plug is reduced because of bypass flow.

## I. INTRODUCTION

The objectives of the SNAP-8 Materials Program are to provide data to serve as a basis for the selection of materials for use in various components; to assist, through metallurgical studies, in the design, development, fabrication, and testing of the SNAP-8 system; and to evaluate the resistance of the reference material (i.e., 9Cr-1Mo alloy steel) to mercury corrosion. This report covers the work performed during the first 6 months of the 1965 calendar year.

## II. COMPONENT DESIGN AND DEVELOPMENT SUPPORT

A high starting torque exhibited by the NaK PMA in water testing during the latter part of 1964 was attributed to a high coefficient of static friction of the NaK-lubricated journal bearing. While the Pump Development Group considered possible bearing redesign, the Materials Section conducted tests to evaluate the possible effect of various bearing-surface conditions. Static friction tests were conducted with 0.950-in.-square hardened-steel (Rockwell C66) inspection-gage blocks. The test results are reported in Ref. 1 (pp. IV-10 and -11). With mating-surface finishes of approximately 1 rms against 1 rms, "wringing" (the effect of the surface tension of the liquid lubricant) resulted in an apparent coefficient of static friction of 0.40.

Molybdenum disulfide most effectively reduced this coefficient (to 0.09), but it will not be effective continuously, because of coating removal in a unit subjected to many starts and stops. The retesting of coated specimens, for example, produced an increase in the measured coefficient of friction to 0.18. A permanent coating of Electrolized chromium (applied by Electrolizing Sales, Inc., Los Angeles) provides greater permanence, at some sacrifice in frictional properties, than molybdenum disulfide. The static coefficient of friction between Electrolized surfaces (25 runs) was approximately 0.16. It was concluded that if the reduction of the static coefficient of friction must be achieved by bearing-surface revision, an Electrolized surface combination appears to be the optimum candidate to provide a significant improvement (approximately 0.16 for Electrolized surfaces vs 0.40 for the bearing surfaces currently in use).

## III. COMPONENT FABRICATION SUPPORT

### A. TUBE-IN-TUBE (T-T) BOILER

The fabrication of a T-T boiler (Figure 1) was initiated during this report period. The first two units are to be coiled to two different diameters (34.5 and 20.5 in.).

#### 1. 9Cr-1Mo Tubing

A production order for 30-ft-long, 9Cr-1Mo steel tubes required for T-T boiler fabrication was completed by Pacific Tube Company, Los Angeles. The Von Karman Center provided the starting stock. This was basically 9Cr-1Mo steel with its chemistry modified to significantly increase the creep and stress-rupture strength above that of the basic alloy (see Ref. 2). Twenty-seven of 38 tubes were rejected for outer-surface defects on in-process ultrasonic inspection (see Ref. 3, pp. IV-3 and -4). Twenty-three of the 27 tubes were made usable by grinding out defects that were from 0.003 to 0.020 in. deep (predominantly 0.006 in.). Metallographic study indicated that the defects were probably caused by excessive inclusions in the starting raw material and that the possible presence of these defects (undetectable on final inspection) means that the performance of this tubing is unpredictable. A decision was made to use the tubing for boiler fabrication, because it appears that if a leak develops during boiler operation it will probably be very small initially, due to the alignment of individual inclusions parallel to the tube surface. Methods are available for detecting small through-the-wall leaks, should they occur, and are being considered for incorporation in the various SNAP-8 test systems.

A cleaning procedure - Type VI in the SNAP-8, 9Cr-1Mo steel, cleaning specification (Ref. 4) - was devised and successfully used for removing a tightly adherent surface oxide film produced during the final heat treatment of the 9Cr-1Mo steel tubes (in an exothermic gas atmosphere). It employs an alkaline permanganate dip, followed by immersion in a nitric-hydrofluoric acid bath, and finally cleaning with a steam spray.

### 2. Development of Fabrication Processes

Experiments were conducted by Cromer Processing Company, Long Beach, California and Cooney's Pipe and Copper Works, Wilmington, California, subcontractors of Western Way Manufacturing Company, Van Nuys, California (SNAP-8 contractor for T-T boiler fabrication), on procedures for coiling the boiler with various internal-tube-reinforcement materials. Sample coils were bent with no reinforcement and with tree-resin or sodium thiosulfate (hypo) reinforcement material. It was determined that tree resin was required in the annulus formed between the external surface of the mercury-containment tubes and the internal surface of the outer 321 SS tube. The interior of the 9Cr-1Mo mercury-containment tubes, it was found, required no reinforcement when the 34.5-in.-dia tube assembly was bent, but tree-resin reinforcement was required, to avoid excessive tube collapse, when the 20.5-in.-dia assembly was bent. Cleaning procedures were established at the Von Karman Center through support of the fabricators' experimental program in order to completely remove the tree resin after boiler coiling. Double-cycle cleaning with an alkaline solution (MIL-C-14460, Type I) proved adequate. The experimental effort and the successful procedures established for T-T boiler cleaning are described in Ref. 3 (pp. IV-4 and -5) and Ref. 5 (pp. IV-2 and -3).

## B. SPECIFICATIONS

The following specifications were issued or amended during this report period:

| No. | Title |
| --- | --- |
| AGC-10319/2 | Degreasing of SNAP-8 Nonprecision Components and Systems, Procedure for |
| AGC-10319/4 | Cleaning of SNAP-8 Non-mercury Test Support Subassemblies, Workhorse Equipment, and Raw Materials, Procedure for |
| AGC-10319/3 | Acid Pickling of Nonprecision SNAP-8 Component Parts, Procedure for |
| AGC-10319/6 | Cleaning of SNAP-8 Power Conversion System Components Prior to Final Assembly, Procedure for |
| AGC-10197 | Alloy Steel, Sheet, Strip, Bar, Plate and Forging (9 Chromium - 1 Molybdenum) |
| AGC-10226 | Steel, Cleaning and Dehydration, Procedure for |

## IV. TEST OPERATIONS SUPPORT

### A. RATED POWER LOOP-2 (RPL-2)

This loop is being operated to evaluate the performance of various SNAP-8 components, including the mercury boiler, TAA, condenser, and mercury PMA. A test series was completed during November-December 1964 with rubidium additions in the mercury. Three operating runs were completed during this report period. In February 1965 the loop was operated with no Rb additive. Prior to this run the mercury inventory was changed and the mercury dump tank was cleaned. In March a single Rb injection (1180 ppm) was made at the start of a test run, and the loop operated with no further additions until it was shut down (because of apparent degradation of boiler performance, as discussed below). After the mercury inventory was changed and the dump tank was cleaned, the loop was operated for a final period during May and June with no Rb additive.

#### 1. Mercury Loop

##### a. Boiler Cleaning

Prior to loop operation in February with no Rb in the mercury, the RPL-2 boiler was cleaned to remove any residual contamination from the previous operating period that could potentially inhibit proper boiler conditioning when RPL-2 operation was resumed. The potential contaminants are Rb oxides, internal-surface scale, or silicone-oil decomposition products. The boiler was cleaned by an outside contractor (Consolidated-American Services, Inc., Hawthorne, California), using the procedure described as Method II in Aerojet Specification AGC-10319/6. The drying was performed by flushing with hot nitrogen until a dewpoint of -40°F was achieved. The nitrogen used was from a liquid nitrogen source that was free of impurities, as determined by gas chromatography.

b. Rubidium Additions

As the RPL-2 boiler accumulated operating time, the boiler performance became less dependent on the presence of the additive Rb. The performance during November-December 1964, when the first run was made with Rb, is described in Ref. 6. This performance was unpredictable, and periodic additions were required to maintain the proper boiler heat transfer. The test in February 1965, during which no Rb was added, lasted approximately 100 hours. Prior to the start, the boiler was alkaline-cleaned (using the procedure described in Aerojet Specification AGC-10319/6), the mercury inventory was replaced, and the mercury dump tank was cleaned of all traces of Rb and/or its oxides. The boiler did not achieve the rated mercury-output conditions. The next test run in March, which included Rb additive to achieve proper boiler performance, lasted more than 100 hours. Contrary to the experience in the November-December 1964 test (see Ref. 5, p. IV-3), only a single injection of Rb was sufficient to maintain satisfactory boiler performance throughout the run, which included three shutdowns. Although Rb loss apparently occurred during each shutdown, the remaining Rb was sufficient for boiler surface conditioning. The May-June 1965 test run was conducted with no Rb additive, and it was found that none was required for satisfactory boiler performance. To ensure the absence of Rb in solution at the start, the mercury inventory was changed and the mercury dump tank was alkaline-cleaned (as it had been prior to the February test).

It is suggested that the Rb produces wetting between the mercury and the tube wall and that this facilitates heat transfer. Without Rb but with sufficient operating time, as had been noted previously, the boiler surface becomes "conditioned." An additive is not required to ultimately achieve proper boiler performance. As this RPL-2 boiler accumulated more operating hours the mercury surface thus became conditioned and the boiler was able to perform properly without the additive. The Rb served as a conditioning agent during what would otherwise have been a nonperforming run-in period. The hours accumulated up to the start of the May-June test apparently produced the necessary conditioned surface, and the result was satisfactory boiler performance, with no need for Rb additive.

After the RPL-2 mercury boiler started operating satisfactorily without Rb, periodic residue and mercury samples were taken from the loop during shutdowns. It was found that the mercury remained free of Rb but that residues from piping, components, and the surface of the mercury in the dump tank contained traces of Rb, probably in the form of an oxide. The Rb depleted from the mercury during previous runs apparently was removed by oxidation, due to air exposure during component replacement or to oxidizing impurities in the argon-cover-gas system of the mercury loop. The Rb oxide was deposited on piping or component surfaces or was washed into the dump tank during subsequent mercury dumps. Apparently, this material cannot readily be completely washed off a metal surface; it has been continually detected, in ever-decreasing amounts, in all residue samples analyzed during this report period. Reports on Rb analyses are presented in Refs. 1 (p. IV-8), 3 (pp. IV-6 and -7), 5 (p. IV-3), and 7 (pp. IV-1 and -2).

c. Organic-Fluid Contamination

Analyses of residue samples periodically removed from mercury-loop components indicate that organic materials are entering the loop. Mix-4P3E was detected, as were various oils used in vacuum pumps in the system. An investigation was conducted to determine potential sources of these organics; the results are presented in Ref. 7 (pp. IV-2 and -3).

The specific source of these fluids is not apparent at present. A danger exists, however, that they could be transported to and decondition a satisfactorily operating boiler. Capsule tests conducted by Dr. L. Rosenblum at the NASA Lewis Research Center indicate that even a very thin surface oil film may prevent proper heat transfer from the tube wall to the mercury stream.

d. Particulate-Matter Contamination

Residue samples containing metallic elements have periodically been removed from RPL-2. The residues varied in quantity, particle size, and chemical composition. Deposits not affecting loop operation were found during this report period; their chemical composition indicated mercury corrosion, and they were of extremely small particle size. However, instances of component-performance degradation did occur because sufficiently large metallic particles were present to clog small mercury-flow passages. It appears that particulate matter, whatever its origin, is being circulated through the loop by the mercury. Because larger-particle transfer could impair performance, filter screens were incorporated upstream of critical components to minimize flow-passage clogging.

(1) Plug-In Boiler-Tube-Inlet Restrictor Orifice

After the March test period, the boiler-inlet manifold was removed. A particle was found lodged in the upper-left tube-inlet-restrictor orifice (see Figure 2). Evaluation indicated that the particle was of mild steel and probably was a machining chip (see Ref. 5, p. IV-3). Potential loop sources for this chip are the turbine-simulator lines and some mild-steel parts of the boiler.

(2) Mercury Pump-Motor Assembly

After the March test period, partial disassembly of the mercury PMA revealed that solid particulate material had clogged the jet-pump orifice; this material looked like the residues previously removed from the loop. Chemical analysis and metallographic examination (see Ref. 5, p. IV-5) indicated a mixture of metallic chip-like particles and weld spatter. One weld-spatter particle consisted of 316 SS. The remaining weld spatter and the magnetic chips were of an iron-base material, but the analysis did not definitely establish the alloy. One nonmagnetic particle consisted of Stellite 6B; two valves in the system contain Stellite 6B, as does the turbine assembly. An analysis indicated that this particle could be part of the trailing edge of a turbine nozzle.

(3) Turbine

A turbine-pressure transducer was removed for calibration during a loop shutdown. A wet brown residue was found in the 1/4-in. loop standoff line to the transducer. Qualitative analysis indicated that the residue contained approximately 17% Rb, probably in the form of an oxide; the balance was primarily Fe, with a significant quantity of Ni and minor amounts of Mg, Si, and Cu. The function and calibration of the transducer had not been affected. It was concluded that the residue consisted of finely divided metallic oxides similar to those previously found in the mercury loop. Apparently they had been carried by the mercury vapor from the boiler and deposited in the pressure-transducer line.

## 2. NaK Primary Loop

Various pipe sections and components of the RPL-2 NaK primary loop were analyzed and evaluated during this report period. Some analyses were conducted as part of a continuous evaluation of loop-operation effects, and others because of component failure. All evaluated components had operated in the loop before the later-1964 incorporation and use of a NaK-purification system. A marked improvement in NaK-primary-loop oxygen control occurred subsequently, as evidenced by the absence, toward the end of this report period, of observable component degradation due to mass-transfer buildup.

a. NaK-Flow Venturi

The throat section of the NaK-flow venturi was examined in January 1965 after approximately 800 hours of operation. Most of this time had been accumulated in the loop during operation without oxygen control. Water-flow tests indicated that the venturi readings would imply flow rates 25% higher than the actual rates. The inlet and throat contained mass-transfer deposits; the outlet contained a much smaller amount of deposited material. The deposit had flaked off the venturi wall in local areas of the throat. The remainder of the deposit was tenacious but could be scraped from the walls. A sample of the residue, analyzed qualitatively by emission spectroscopy, was found to contain the approximate ratios of chromium, nickel, and molybdenum found in 316 SS (17 to 12 to 2.5, respectively). Although the residue was principally magnetic, some nonmagnetic residue was found.

b. NaK-Heater Connection Piping

Sections of the loop piping at the gas-fired NaK-heater inlet and outlet were examined during January; their respective operating temperatures are 1100 and 1300°F. The inlet was coated with a thin, grayish-black, crystalline deposit. The outlet contained no deposits and had a matte finish of silver-gray with a metallic luster. The microstructure of the inlet section indicated a surface deposit and an intergranular deposit extending approximately 0.001 in. cross sectionally into the pipe. The microstructure of the outlet piping indicated the possible presence of minor deposits, but intergranular penetration was not evident.

c. Electromagnetic Pump

Mass-transfer deposits were found in the pumping section of the primary NaK EM pump that was removed from the loop at the end of 1964. The deposits resulted in reduced NaK flow through the pump at the rated current flow. They were hard and tenacious and appeared to be concentrated in bus-bar-attachment areas. The pump was returned to the vendor (Mine Safety Appliances Company) for examination, with a request that the throat section of the NaK pipe be returned for Aerojet evaluation.

d. NaK-to-NaK Heat Exchanger

An analysis was performed on the 316 SS, NaK-to-NaK, heat exchanger that failed after 1356 hours and 47 cycles of primary-loop (PL) operation and 1170 hours and 44 cycles of heat-rejection-loop (HRL) operation. The failure occurred at the outlet end of the heat exchanger.

The 316 SS heat-exchanger material was not detrimentally affected by the service conditions. However, the operational stresses imposed on the heat exchanger at the weld connecting it to the heavy-walled loop piping apparently exceeded the ultimate strength of the thin exchanger-shell material, resulting in component failure. Evidence was found that both metal mass transfer and carbon transport had occurred. The mass transport is assumed to have occurred during previous loop operation without the NaK-purification system. According to Oak Ridge National Laboratory results (Ref. 8), the source of carbon transported to the 316 SS in this system is the NaK-exposed surface of the 9Cr-1Mo steel, mercury-boiler tube. A detailed report on the analysis of this component is presented in Ref. 7 (pp. IV-3 and -4).

Following tests in RPL-2, the boiler will be dismantled and evaluated to determine the extent of decarburization. The effect of such decarburization on the creep and rupture strength of 9Cr-1Mo steel at approximately the maximum boiler-operation temperature (1300°F) is being evaluated in pressurized-capsule tests at Aerojet-General Nucleonics (see Ref. 9, pp. VIII-20 to -28).

e. Deterioration of Heat-Transfer Cement

A graphite-base, heat-transfer cement (Thermon Type T-63 produced by Thermon Manufacturing Company, Houston, Texas) had been applied to provide a heat-transfer medium between trace heaters and the RPL-2 liquid-metal lines. Examination after the March operating period indicated that the cement had degraded on several lines (see Ref. 3, pp. IV-5 and -6). In one instance, the cement temperature was below the maximum use temperature specified by the vendor. In another instance, improper control of NaK-line heaters raised the cement temperature significantly above the specified maximum of 1250°F. The degradation resulted from graphite oxidation and melting of the cement binder adjacent to the heater sheaths. The heat-transfer cement was thus converted into an insulating material. No evidence of cement degradation was apparent on lines maintained at 600°F or lower. The cement was removed from the high-temperature lines to prevent possible trace-heater (and perhaps liquid-metal-pipe) failures. Temperature-control instrumentation was incorporated in the trace-heating-element circuits to limit the maximum sheath temperatures.

f. Cleaning of Transducers

A technique was developed to clean transducers that become contaminated and/or clogged with a mixture of NaK and oxides (see Ref. 10, p. IV-6). The requirement for cleaning resulted from the clogging of RPL-2 transducers, which occurred because the loop was operated without the NaK-purification system. The temperature limitation of transducers requires a temperature-reducing standoff-leg connection between the loop and the transducer. This standoff leg acted as a natural cold trap for the NaK loop, leading to the precipitation of oxides.

A procedure was also established for the cleaning of oil-contaminated transducers (Ref. 1, p. IV-10).

g. Compatibility Between Indium and Thermocouple-Well Materials

Indium is employed in thermocouple wells in the SNAP-8 loop as a heat-transfer medium between the well and the thermocouple sheath. Tests were conducted to evaluate the compatibility between indium and 410 and 316 SS (the thermowell and sheath materials). Sealed isothermal capsules were exposed for 2000 and 2500 hours at 1300°F. Metallographic examination indicated that the 410 SS and 316 SS had not corroded.

3. Lubricant/Coolant Loop

Precipitated crystals were found in a mix-4P3E sample from the first order filled by Shell Development Company, Emeryville, California. This precipitation should not have occurred. A sample of the decanted liquid and the crystals were analyzed by gas chromatography. The results, shown in Table 1, were compared with (a) the original vendor analysis, supplied with the delivered fluid, and (b) the average of several recheck analyses performed at the Von Karman Center (VKC) on the as-received fluid. The precipitate was found to contain a comparatively high amount of the p-p isomer. A discussion with Shell chemists led to a conclusion that precipitation resulted from an excessively high content of p-p isomer in the original mix-4P3E fluid. The critical amount appears to be 3%. A VKC recheck analysis of the fluid confirmed that the p-p isomer content was greater than 3% (see Table 1). A subsequent shipment of fluid was analyzed, and the p-p isomer content was found to be satisfactory (i.e., below 3%).

Aerojet component testing with mix-4P3E produced data on the dynamic viscosity of the fluid (the constant of proportionality between an applied stress and the resulting shear velocity) that indicated mix-4P3E might not be a true Newtonian liquid. At Aerojet's request, Shell Development ran dynamic tests with a Hoake Rotovisco (rotary viscometer). Tests at 68, 122, and 176°F indicated that the mix-4P3E is indeed Newtonian in its behavior. Typical test results are presented in Ref. 1 (pp. IV-11 and -12).

A sample of the mix-4P3E removed from RPL-2 after the March operating period was analyzed (see Ref. 5, p. IV-5). This examination, following approximately 800 hours of testing, showed very little change in physical properties (which should not affect fluid usability), but the fluid was discolored. The discoloration appeared to have been caused by products of the thermal decomposition of mix-4P3E. A carbonaceous deposit on the Calrod heater sheath indicated that the fluid in contact with the heater surfaces had been heated above the mix-4P3E decomposition temperature, which is 800°F.

4. NaK-Purification System

A pipe failure occurred in the HRL purification-system line during this report period. The 316 SS pipe material did not cause the failure, which rather appeared to be associated with a trace heater used to maintain the pipe temperature during loop operation. Five holes through the pipe wall had been produced in the failure area within an 8-in. pipe length. The microstructure indicated that the 316 SS had been molten in the area of each hole. A NaK fire that resulted from the failure prevented complete evaluation. Subsequent examination of failed trace heaters (see paragraph IV,B,2,a below) indicated, however, that electrical shorting of the heater through the pipe to ground may have caused both failures.

B. SYSTEM LOOP TEST FACILITY 1 (SL-1)

1. Fabrication

A theory was developed, based on LeRC mercury-capsule work, that an oil film on the mercury-tube wall will prevent proper boiler conditioning. It was reported that these capsule tests illustrate that oil films as little as 6 angstroms thick (approximately $2 \times 10^{-8}$ in.) could prevent proper heat transfer from the tube into the flowing mercury. The PCS-1 system was therefore cleaned, prior to final assembly, to remove oil residue from all surfaces. The entire system was cleaned on an assumption that oil present in any part of it could be carried to the boiler by the mercury and result in boiler deconditioning. Two alkaline cleaning solutions were used: Cee-Bee Chemical Company, Inc. (Downey, California) MX-12 alkaline rust remover (approximately 70% sodium hydroxide), and Alconox (an alkaline detergent, Standard Scientific Supply Corporation, New York, N.Y.). The TAA and mercury PMA were not cleaned in this fashion, but individual parts were ultrasonically cleaned in Freon during assembly.

Defects were radiographically detected on the inner surface of 9Cr-1Mo steel tubing procured (to ASTM-A-213 Grade T9) for mercury-containment tubes of PCS-1. Metallographic examination revealed that these lap-type defects introduced a possibility that metal slivers would be removed during loop operation. All 9Cr-1Mo steel, PCS-1, mercury-loop tubing was X-rayed, and the sections containing defects of this type were replaced.

## 2. Evaluation and Analysis of Loop Sections

### a. Trace Heaters

Sections of three trace heaters removed from the L/C line of SL-1 were evaluated after two of them failed. It was found that (1) one had broken and the sheath and melted in the area of the break, (2) another contained a broken Nichrome heating element (the sheath appeared intact), and (3) the other had a significant increase in electrical resistance (from 18 ohms to 26 ohms). Metallographic and radiographic examination indicated that the first two heaters failed because the Nichrome heating wire melted due to overheating (see Ref. 5, p. IV-5). The molten wire had flowed through cracks in the magnesium oxide insulation and shorted the heating wire to the sheath. It is presumed that the cracks were produced when the heaters were bent to fit the loop-pipe contour. The apparent primary cause of the overheating was the unintentional presence of thermal insulating material between the heater and the pipe in localized areas. The trace-heater installation procedure was changed so that thermal insulation was kept from critical areas of the assembly. The incorporation of temperature-control instrumentation, and/or change in the location of control thermocouples, should minimize the danger of overheating.

### b. NaK Heat-Rejection-Loop Chempump

The HRL Chempump (manufactured by the Chempump Division of Fostoria Corporation) failed after a few seconds of rotation during a wet shakedown test conducted in May. Three samples were collected while the pump was being disassembled: (1) A sample of liquid NaK was drained from the housing, (2) a flake-like particle (3/4 by 1-1/4 in.) was removed from the pump housing, and (3) residue scrapings were obtained from the bearing housing. The three samples were treated with alcohol and were water-washed to remove NaK or NaK oxide. The residue recovered from the NaK sample appeared to be a mixture of very fine, multicolored particles. A portion of this residue, apparently black in color, was soluble in the water and is presumed to have consisted of finely divided oxides of NaK. Microscopic examination of the insoluble residues from the housing scrapings and from the liquid NaK removed from the housing revealed they were composed of fine magnetic and nonmagnetic metallic particles and non-metallic particles that were white, tan, red, and transparent. The particles were too small for individual analysis. A combined qualitative analysis by emission spectroscopy indicated significant quantities of Fe, Ni, Cr, Mo, Al, Mn, and Si. The metallic particles apparently resulted from pump-bearing galling that occurred during the failure. The nonmetallic particles, according to their appearance and the composite analysis, could have represented (1) an abrasive used for powder blasting, or (2) residue from a grinding operation. Their presence would be explained by incomplete removal after some pump component was exposed to such an operation prior to assembly. Microscopic examination and infrared-absorption analysis of the flake-like particle indicated it was a polymeric material similar to polyvinylidene chloride. Its source is not apparent at this time.

## C. LIQUID-NaK LOOP 3 (LNL-3)

### 1. Loop and Component Cleaning

The SNAP-8 LNL-3 system is being operated to evaluate the performance characteristics of the NaK PMA. At the beginning of this report period the loop had operated with no oxygen-control procedure. An in-line cold trap was subsequently incorporated. While the loop was being operated with a probable high oxygen content, the loop and several pumps undergoing testing required internal flushing to remove oxide buildup. The first pump tested was disassembled in an oil bath and the oxide buildup was confirmed. Two subsequent pumps and the loop were flushed without disassembly. The procedure (Ref. 7, pp. IV-5 and -6) utilizes alcohol and water as flushing media. It permits the NaK PMA to be cleaned while it is installed in a loop, and avoids time-consuming and costly teardown and reassembly to restore the unit to an operable condition.

A black residue was found in the cleaning fluids when the last pump (PMA Part No. 093200-13A, Serial No. A-1) was cleaned by internal flushing. Infrared spectrophotometry produced a representative curve that was not identifiable. A review of the assembly and operation procedures indicated that one, or a combination of, five organic materials could have been the source of the residue: (a) Duoseal vacuum-pump oil, (b) mix-4P3E, (c) DC-703 silicone oil, (d) MIL-L-644 preservative oil, and (e) Freon-TF. These materials will be reacted with NaK at 500°F, and the infrared-analysis curve for the resultant residue will be compared with the curve for the residue found in the cleaning fluid.

### 2. Component Analysis

Two NaK PMA housings - Part No. 093247, Serial Nos. A-1 and A-5 - were evaluated (see Ref. 3, pp. IV-8 and -9) after NaK leaks developed during operation tests. It was found that the castings had excessive porosity and, in one instance, a casting cold-shut resulted in an unfused through-wall plug. It was concluded that the NaK leaks were caused by casting defects.

On the assumption that porosity defects undetectable by nondestructive-test techniques may exist in castings, two courses of action were pursued: (a) Because castings appear to be unreliable for NaK service, housings are being fabricated or wrought material, and (b) it was decided to evaluate the brush plating of the inner cast-housing surface with iron to provide a seal against NaK through-wall leakage so that testing could be continued until replacement housings were fabricated. The brush plating is being done by Brooktronics Engineering Corporation, North Hollywood, California. A housing that had previously failed due to through-wall leakage was brush-plated and returned to testing; through-wall leakage again occurred. This housing will be metallurgically evaluated when testing has been completed.

## D. LIQUID-MERCURY LOOP 3 (LML-3)

The SNAP-8 LML-3 system is being operated to evaluate the performance characteristics of the mercury PMA. The Serial No. A-1 pump was removed from testing and disassembled after 120 hours of operation. Various pump components that appeared to have been affected during operation were evaluated (Ref. 10, pp. IV-3 and -4).

A paste-like Cerrobend* residue was found in the visco pump seal, and a black, flaky, residue (believed to be iron-base-alloy surface scale and/or machine chips) was found in the mix-4P3E fluid.

The LML-3 system was apparently assembled in a manner that did not assure overall cleanliness. The observed contaminants, which could cause the abortion of an otherwise successful test, were transported from some part of the system by the mercury and mix-4P3E into an originally "sterile" critical component, the mercury PMA.

## V. TRANSFORMER-REACTOR ASSEMBLY, HEAT-SINK TRANSITION JOINT

The objective of this task is to establish an adequate joint for connecting the heat sink of the transformer-reactor assembly to the line of the L/C loop. The heat sink is fabricated of pure aluminum and the L/C-loop piping of 304 SS.

### A. JOINT CONCEPT

The direct welding of aluminum to stainless steel is not an established state-of-the-art procedure. Several companies are working on the problem. Nuclear Metals, Inc. (NMI), Concord, Massachusetts provided samples of a hot-coextruded transition joint for evaluation. An acceptable transition joint using state-of-the-art procedures was made by joining aluminum to copper by means of the Koldweld (pressure-welding) process of Kelsey-Hayes Company, then joining copper to nickel by TIG welding, and then joining nickel to 304 SS by TIG welding. Previously prepared lap and butt weld samples had indicated (Ref. 11, pp. VIII-1 to -3) that the copper end of the cold-welded part should not be welded directly to the 304 SS. It was concluded that a nickel transition piece is needed between the copper and the 304 SS.

There is evidence that copper is attacked by the SNAP-8 L/C fluid (mix-4P3E polyphenyl ether) if the system operates under nuclear radiation (see Ref. 12). Because of the potential copper-corrosion problem, it is necessary to apply a protective metallic coating to keep copper from contacting mix-4P3E.

### B. PROTECTION OF COPPER IN ALUMINUM-COPPER TRANSITION JOINT

A test program to evaluate three optimum candidate coatings (electroless nickel, silver over electroless nickel, and Electrolized chromium) was completed during this report period (Ref. 13). Coated specimens were exposed (for up to 2500 hours) to mix-4P3E heated to 250°F in evacuated glass capsules ($10^{-3}$ to $10^{-4}$ torr).

Metallographic examination of specimens coated with electroless nickel and silver over electroless nickel revealed a reaction between the aluminum portion of the transition joint and the nickel plating. The reaction is

*A low-melting alloy used for internal support of LML-3 lines when bending was required during loop assembly.

believed to have been galvanic corrosion that occurred during specimen plating. This attack is considered detrimental to reliable protection of the copper portion of the joint and eliminates any coating system employing electroless nickel on aluminum.

Metallographic examination of specimens coated with Electrolized chromium revealed satisfactory adherence, but coating voids were found on the unexposed specimen and coating cracks were found on the exposed specimens. These coating defects contraindicate the use of Electrolized chromium unless additional tests show that they can be avoided in plating and that subsequent exposure does not, in fact, detrimentally affect the layer. There are no plans for additional testing, because the transformer-reactor assembly development task has been curtailed. Since copper is resistant to the L/C fluid in the absence of radiation, the near-future tests of the SNAP-8 system need not rely on a coated transition joint.

### C. DIRECT-BONDED ALUMINUM-TO-304 SS JOINT

An evaluation of experimental specimens of direct bonding between aluminum and 304 SS was also completed. These specimens were made from a tubular transition joint furnished by NMI and were exposed to the same test conditions as the coated four-metal transition joints. The direct-bonded specimens appeared unaffected by the exposure conditions. Metallographic examination showed no surface reaction with the test fluids, mix-4P3E or mix-4P3E with water (see Figure 3). In mix-4P3E the specimen weight changed in such a manner that, if it is assumed the total attack was confined to the aluminum (the lighter of the two metals), the exposed surface was dissolved to a depth of $0.2 \times 10^{-5}$ in. Assuming that only the 304 SS (the denser material) was attacked, the conversion of the weight change to depth of penetration results in even less surface penetration. In mix-4P3E + 1 vol% $H_2O$, a positive specimen-weight change occurred but was so slight that metallographic examination did not reveal a surface reaction. Additionally, no evident cross-bond diffusion resulted from the 2500-hour exposure of the direct-bonded Al-to-304 SS specimen at the 250°F test temperature.

This joint should be considered further as an alternative to the coated four-metal transition joints discussed above. Because it represents an advance in the state of the art (the four-metal transition joint represents available state-of-the-art procedures), further testing of environmental effects would be required. Such testing is not planned, because of the decision to curtail transformer-reactor assembly fabrication and testing.

## VI. BIMETAL-TUBE EVALUATION

### A. 9Cr-1Mo/316 SS TUBE

The objective of this task is to establish the suitability of a bimetal tube comprised of nonrefractory, conventional materials as a backup to the 9Cr-1Mo steel, mercury-containment tube for boiler service.

A 9Cr-1Mo steel/316 SS tube butt-weld joint specimen was prepared by manual TIG welding and was evaluated (Ref. 1, pp. IV-12 and -13). The abutted

9Cr-1Mo steel liner was first welded in an area where the 316 SS had been removed by machining. The joint was then completed by filling the void in the 316 SS clad, over the 9Cr-1Mo steel weld, using 316 SS filler material. Metallographic examination of sample sections indicated the retention of individual layers of 9Cr-1Mo steel and 316 SS in the welded area; however, the 9Cr-1Mo steel liner collapsed. The collapse may have occurred during welding, because of the stress imposed on the thin wall (0.020 in.) by the higher thermal contraction of the 316 SS. Additional samples are to be prepared to ascertain if a change in welding techniques will eliminate this condition or minimize it to within acceptable limits.

### B. DEVELOPMENT OF REFRACTORY-BIMETAL TUBING

The objective of this task is to establish the availability of refractory-bimetal tubing as a backup for mercury containment in the SNAP-8 system if experience indicates that 9Cr-1Mo steel (the present reference material) does not exhibit sufficient mercury-corrosion resistance for a life of 10,000 hours. If direct-bonded tubing (refractory to 316 SS) cannot be fabricated or proves to be unusable because of high-temperature diffusion effects, a source is to be developed for refractory-bimetal tubing fabricated with an interface material, or materials, between the refractory and the 316 SS.

An order for columbium/316 SS tubing was completed by NMI. The technical requirements for the tubing are covered by Engineering Data Sheet No. 390-64-0188B (see Appendix A of Ref. 14). Twelve tube lengths of two different sizes (0.400- and 0.684-in. ID by 0.055-in. wall, of which 0.020 in. was Cb) were received and evaluated (Ref. 1, pp. IV-13 to -15). Ultrasonic inspection and confirming metallographic examination (see Figures 4 and 5) indicated that only three lengths (none of the large ID) contained an acceptable metallurgical bond between the Cb and the 316 SS. There is no intention at present to pursue the development of a refractory-bimetal tube as part of the SNAP-8 program at Aerojet.

## VII. BOILER CONDITIONING STUDIES

The objectives of this task are (a) to evaluate the effect of various tube-surface conditions on the capacity of the SNAP-8 boiler to produce a superheated mercury vapor on system startup, and (b) to establish procedures for removing any surface residue found to be detrimental to boiler conditioning.

### A. MIX-4P3E RESIDUE REMOVAL

There is a potential danger that mix-4P3E may enter the mercury system through the various L/C-fluid, mercury interfaces. Selected tests were performed to evaluate methods of removing this contaminant if intrusion into the boiler does occur and if the fluid is thermally decomposed on the mercury-tube wall.

After preliminary tests, a program was conducted to evaluate several of the better resultant candidate methods for removing thermally decomposed mix-4P3E from a 9Cr-1Mo steel surface. It was established that the surface can be cleaned by a double-cycle procedure (Ref. 3, p. IV-9).

The residue is first oxidized in an air atmosphere at 1300°F. The surface is then solution-cleaned with Turco Products, Inc. fluids as follows: Turco 4931 followed by Turco 4338C and finally by Turco 4931 again. The experimental effort will be documented by a Technical Memorandum.

B. LOOP TESTS

A task was started in March 1965 to reactivate Component Test Loop 2 (CTL-2) and investigate the effect on boiler performance created by adding rubidium to the mercury. Its objectives are to determine (1) the effect of the SNAP-8 L/C fluid (mix-4P3E) on boiler performance, (2) the amount of Rb necessary to improve the performance of the deconditioned boiler, and (3) the amount of Rb lost during an extended loop run. An apparatus was designed and fabricated for making controlled Rb additions or a mix-4P3E addition to the mercury in CTL-2.

1. Test-Loop Modifications

a. Bypass for Mix-4P3E Addition

To provide the necessary boiler deconditioning prior to Rb additions, a valved capsule was installed in parallel with the mercury-pump bypass. Figure 6 shows the capsule installation schematically. The capsule was loaded with mercury and 25 g of mix-4P3E.

b. Rubidium-Addition System

Because the quantity of Rb required to condition the boiler was unknown, an addition system was designed that allows an unlimited quantity of Rb to be metered into the mercury.

Figures 6 and 7 show the system. The stainless-steel addition tank was atmospherically sealed. Mercury from the loop was allowed to circulate through the tank by way of the paralleled bypass lines. The mercury level in the tank was controlled by means of pressurized-nitrogen cover gas. When Rb was to be added, circulation through the tank was valved off and the tank was evacuated. Under vacuum, the 1% Rb-Hg solution (10 lb of Hg to 0.1 lb of Rb) was metered into the tank, the cover gas was reapplied, and circulation was resumed. After 1/2 hour of circulation through the loop, a mercury sample was drawn from the tank and was analyzed by titration to determine the Rb concentration. A second sample was taken and analyzed just before Rb additions. If no Rb was added, mercury samples were taken at least twice daily for Rb analysis.

2. Operation

a. Run 1 (Base Run)

The mercury in CTL-2 was replaced and the base run was initiated on 14 April 1965. The loop operated for approximately 5 hours, when a mercury-pump electrical problem caused a shutdown. The pump was replaced, the base run was resumed, and the loop was brought to the rated SNAP-8 conditions at the boiler outlet (265 psia at 1265°F) after approximately 300 hours of operation. Before this run, CTL-2 had been used for conditioning experiments that included oil additions to the boiler and for various cleaning experiments (Ref. 15, p. 6). The extended run-in time to condition the boiler had not been expected.

b. Run 2 (Mix-4P3E and Rubidium Additions)

The loop was started again on 3 June 1965, was brought up to the rated conditions on 7 June 1965, and 25 g of mix-4P3E was admitted to the mercury-pump suction. An effect on boiler condition was immediately apparent, and within 45 min the boiler-outlet temperature had dropped from 1270°F to 1100°F, indicating that the boiler was deconditioned.

Rubidium additions were started on 8 June 1965. Small amounts were added to the mercury in the loop through the addition tank. The concentration was determined periodically in the manner described above. Partial conditioning (boiler-outlet condition, 1270°F and 165 psig) was indicated after the Rb concentration reached 600 ppm; this could not be sustained when the boiler-outlet pressure was increased, as indicated by a decrease in the boiler-outlet temperature. The Rb content of the mercury appears to decay if Rb is not added constantly. Boiler-performance and Rb-analysis data plotted in Figure 8 show the boiler-outlet temperature following a trend similar to that of the relative Rb concentrations in the loop.

The Rb additions will be continued until the boiler performance is restored and sustained at the SNAP-8 rated condition (265 psia at 1265°F).

## VIII. 9Cr-1Mo STEEL, STRENGTH EVALUATION

The objective of this task is to evaluate the strength of various weld configurations in the SNAP-8 system where at least one component is 9Cr-1Mo steel, and the potential creep of SNAP-8 boilers.

### A. WELDS

Eight sheet specimens were tested in air at 1325°F for up to 3000 hours as outlined in Ref. 15 (p. 24). The results of these tests of welds between specimens of 9Cr-1Mo steel are reported in Ref. 16. In summary, they indicate that welding does not decrease the creep or creep-rupture strength of 9Cr-1Mo steel at 1325°F.

### B. ENVIRONMENTAL CYCLIC CREEP

Cyclic-creep testing was undertaken to determine whether excessive creep of the existing SNAP-8 boilers can be expected in the desired 10,000-hour lifetime under the environmental and operating conditions of the boilers.

A trial run to ensure that the desired decarburizing conditions will be achieved by the apparatus was described in Ref. 15 (p. 26). The cyclic-creep testing was conducted and the results were evaluated during this report period. A summary follows.

## 1. Experimental Approach

Three 9Cr-1Mo steel capsules (Figure 9) were stressed in a NaK environment at 1325°F to simulate the SNAP-8 boiler environment. Stresses were induced by pressurizing the internal NaK. Thin sections were machined in the walls to control the stress location and to permit two different stresses to be investigated in each capsule. The pressure was changed each 30 sec to achieve a ±10% stress cycle. The temperature was not cycled, because its effect could be predicted.

The thicknesses of the walls of the capsule gage sections and the internal pressure were selected to assure that if the NaK and cyclic stress did not adversely affect the material, then one section in each capsule would creep 1% and the other section would creep an amount calculated for the SNAP-8 -1 boiler after (0.3% creep in 10,000 hours). If detrimental effects due to the stress cycling, biaxial stress, NaK environment, or carbon transfer were to occur, they could thus be readily determined by comparing the actual creep with the predicted creep.

The three capsules were immersed in NaK inside a 316 SS capsule (Figure 10), and the assembly was operated in the capsule creep furnace. It was expected that the concurrent exposure of the 316 SS and 9Cr-1Mo steel in NaK would result in carbon transfer from the 9Cr-1Mo steel to the 316 SS. The NaK was stirred electromagnetically so that the carbon-transfer rate would be limited by solid-phase diffusion.

All three capsules were connected to a common pressurized-argon system, and therefore to the same pressure cycle (Figure 11). The test was started with all three capsules under stress, with the intention of removing the stress from one capsule after each of 1000, 1800, and 3000 hours.

## 2. Results

While the capsules were under stress, the temperature was controlled to ±3°F. The pressure cycle and pressure control were maintained within 1% except for periods of minutes when argon bottles were changed. The temperature uniformity along the capsule changed from ±1°F at the start to ±3°F at the end.

Two capsules operated under stress for their scheduled times, but the scheduled 3000-hour capsule ruptured after 7 hours. The ends were cut off the capsules after testing, the gage sections were measured, the bodies were split lengthwise (Figure 12), and samples were cut from the 1%-strain section for micrographic examination. The capsule measurements showed that creep occurred in all sections, as summarized below.

| | | | Results from Biaxially Stressed Capsules | | |
|---|---|---|---|---|---|
| | | | Creep Rate, %/hour | | |
| Capsule No. | Time Under Stress, hours* | Capsule Section** | Planned | Actual | Amount of Increase |
| 1 | 1000 planned (1036 actual) | 1% | $1.0 \times 10^{-3}$ | $3.8 \times 10^{-3}$ | 4 times |
| | | -1 | $3.2 \times 10^{-4}$ | $1.2 \times 10^{-3}$ | 4 times |
| 2 | 1800 planned (1845 actual) | 1% | $5.5 \times 10^{-4}$ | $7.6 \times 10^{-4}$ | 40% |
| | | -1 | $1.75 \times 10^{-4}$ | $5.4 \times 10^{-4}$ | 3 times |
| 3 | 3000 planned (7 actual) | 1% | $3.3 \times 10^{-4}$ | 1 to 3 | 3-1/2 to 4 orders of magnitude |
| | | -1 | $1.04 \times 10^{-4}$ | 0.3 | 3-1/2 orders of magnitude |

Micrographic examination (Figure 12) showed that severe grain growth had occurred, together with elimination of the carbide precipitation in the grain boundaries. A fine dispersion, appearing to be carbides, remained equally distributed throughout the wall, except for a layer less than 1 mil thick on the inside surface. The same precipitation pattern was noted in the unmachined sections of the walls, but some parts of these lower-stressed sections did not recrystallize. The grains in the section that ruptured are 2 to 4 times larger than for the other two capsules, even though the section was stressed the same as the -1 section of Capsule 1. The microstructure is undergoing further study.

No possible operating mistakes could be postulated to account for the high creep rate of Capsule 3, because all capsules were exposed in the same NaK and were open to the same pressure source. The capsules were stress-relieved separately after the 9Cr-1Mo steel was welded to the 316 SS, and this is considered the only operation in which different treatments could have occurred. It is postulated that some unknown factor caused rapid grain growth in Capsule 3 and that, as a result, the creep strength of the recrystallizing material was drastically lowered.

The carbon content of a full wall section cut from the bulged part of Capsule 3 was found to be 0.10%, as compared with 0.15% for the as-received lot.

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* All capsules were exposed in a decarburizing NaK atmosphere at 1325°F for the full length of the test.

** 1% indicates that the capsule section was designed to creep 1% in the planned time; -1 indicates that the section was designed to creep an amount equivalent to that to be expected of the -1 boiler in 10,000 hours (0.3% creep).

## IX. CORROSION-LOOP PROGRAM

The objectives of this AGN program are to determine corrosion and mass-transfer patterns in the mercury and NaK loops of the SNAP-8 system and to evaluate the corrosion resistance of the SNAP-8 reference materials with regard to the 10,000-hour-life requirement.

Corrosion Loops 1 and 2 (CL-1 and -2) were constructed of Haynes 25 alloy. Operation of the first loop was completed in 1962. The second loop was converted into Component Test Loop 2 (CTL-2), which was operated to check the performance of certain components to be used in subsequent loops and to run mercury-boiler conditioning tests (see Section VII above).

The mercury-containment material for CL-3 and -4 was 9Cr-1Mo steel. The NaK primary loop was constructed of 316 SS, with a section of Hastelloy C in the low-temperature area and sections of Chromalized Hastelloy N and 347 SS in the high-temperature area; 316 SS was also used for the NaK-condensing loop.

### A. EVALUATION OF CORROSION LOOP 3

The loop was removed from its test cells and disassembled after a section of 316 SS tubing failed in the NaK heater, causing a shutdown as described in Ref. 15 (p. 31). The components (described in Ref. 17) were disassembled, and all tubing in the loop was split longitudinally with a band saw. The evaluation to date is reported below.

#### 1. NaK Primary Systems

##### a. NaK Heater

Figure 13 shows the failure area in the NaK heater, and Figure 14 shows the microstructure of sections taken from the heater tubing. The failure was caused by intergranular cracking associated with a second phase that precipitated heavily at the grain boundaries, especially in the failure area. Selective etching indicated the precipitate to be sigma phase, but attempts to confirm this by electrolytic extraction and X-ray diffraction have not been successful. No explanation has been determined for the heavy precipitation of the second phase in the failure area and not in other heater areas at equal temperatures. Further efforts will be made to identify the second phase by extraction and X-ray diffraction.

##### b. Mercury Boiler (NaK Side)

The temperature profile of the NaK-boiler shell indicates the temperatures of the 316 SS and the 9Cr-1Mo steel during loop operation (Figure 15). The boiler performance constantly improved during operation, as indicated by a shift in the NaK-temperature profile; the shift stopped after 2000 of the 4400 hours of operation. The boiler temperatures cited in this discussion are taken from the boiler profile after 2000 hours because they represent the highest temperatures to which the materials were exposed.

The 316 SS outer shell of the mercury boiler showed no evidence of corrosion. There was some carburization of the 316 SS in the hottest section (1310°F) of the boiler (Figure 16).

Some exterior cracking of the 9Cr-1Mo steel tubing was observed, primarily in the area of highest heat transfer (Figure 17), and may be associated with thermal stress or thermal fatigue. Tube cracking was also observed on the mercury side (interior) and is discussed in Section IX,A,2 below. Decarburization was found and its depth decreased gradually along the boiler toward the NaK outlet, until a point was reached where the temperature had decreased to approximately 1270°F. No decarburization was observed in tube sections where the NaK temperature was below 1270°F.

c. Other Components and Materials

Other components in the NaK primary system (e.g., the EM pump, EM flowmeter, and 316 SS tubing) showed no indications of corrosion or mass transfer. The sections of Chromalized Hastelloy N, 347 SS, and Hastelloy C incorporated to simulate reactor materials showed no appreciable corrosion (Figure 18). The 347 SS in a high-temperature section of the NaK primary system (1320°F) showed less change in microstructure than the 316 SS.

2. Mercury System

a. Mercury Boiler

The temperatures of the 9Cr-1Mo steel tubing along the mercury boiler are considered to be those given by NaK-temperature profiles. The corrosion found on the mercury side (interior of the tubing) is keyed in this discussion to the boiler profile after 2000 hours of operation.

The boiler-inlet-plug region is 5 ft long. Pitting was found on the 9Cr-1Mo steel plug in an area 7 to 21 in. from the boiler inlet (Figure 19). The inner surface of the 9Cr-1Mo steel tubing in the plug area was lightly pitted. The NaK-temperature profile of the boiler indicates practically no heat transfer in the plug region after the liquid mercury was preheated in the first 2 ft of the boiler. This suggests that the corrosion in the first part of the plug was caused by solution attack until the mercury became saturated. After saturation, no attack on the 9Cr-1Mo steel occurred until higher wall temperatures were encountered farther downstream.

Visual examination of the interior of the 9Cr-1Mo steel tubing following the plug region indicated heavy pitting in some sections. The maximum pitting depths are plotted in Figure 20, and Figure 21 reproduces photographs of boiler-tube sections where pitting was found. The heaviest pitting is associated with the boiler area where the heat transfer from the NaK to the mercury was the greatest, as indicated by the boiler-temperature profile.

The microstructure of the interior of typical sections of the tubing is shown in Figure 22. Tube cracking can again be seen in the boiler area where the maximum heat transfer occurred. Corrosion-product deposition is also shown.

The pattern of corrosion and corrosion-product deposition in the boiler suggests a relationship to the mercury flow pattern or hydrodynamics during the boiling process. It is postulated that the flow consisted of large drops or globules of mercury emanating from the plug section. These drops were forced along at a low velocity and pitted the tubing by solution attack. As drop velocity was increased and drop size was reduced by the increase in quality, the swirl wires in the boiler tube became effective in breaking up the drops and increasing the heat transfer. As shown in Figure 21, at the beginning of the boiler (5 to 15 ft) the pitting was independent of the swirl wire but was predominant in the swirl-wire areas (15 to 28 ft) as droplet velocity increased. It may have been possible for the swirl wires to trap several mercury droplets in the higher-velocity regions, thereby increasing their residence time. In the first 5 to 15 ft of the boiler, the pitting was predominantly in the bottom of the tubing (with respect to gravity), indicating low droplet velocities and swirl-wire ineffectiveness.

Most of the corrosion-product deposition was found 25 to 30 ft from the boiler inlet. As shown in Figure 20, this is the area where the pitting depth decreases sharply. The corrosion products are deposited from supersaturated liquid mercury in the liquid-vapor stream as the vapor quality approaches 100%.

The change in the NaK-temperature profile with operating time indicates that the mercury-flow pattern was changing constantly up to 2000 hours of operation. This would account for the pitting and corrosion-product deposition observed 30 to 50 ft from the mercury inlet. Had the boiler initially performed satisfactorily, producing rated-output vapor at the outset of the test run, it is believed that these effects would have occurred closer to the inlet.

b. Choked Nozzle

The adjustable choked nozzle, which could not be adjusted during the last portion of CL-3 operation, was disassembled (Figure 23) and examined visually. A sheared pin was found in its operating mechanism. The Stellite 6B nozzle and pintle tip were not eroded by the mercury vapor; there was a slight amount of corrosion-product buildup in the nozzle, and the pintle tip and nozzle were wetted by the mercury.

c. Turbine-Simulator Heat Exchanger

No significant evidence of corrosion or mass transfer was found in the turbine-simulator heat exchanger. The tubing appeared to be wetted in some areas.

d. Blade Mockup

The blade-mockup assembly was disassembled, and the blade section was removed for inspection. Figure 24 compares the blade section before and after exposure. The vapor velocity through the blade section was estimated at 206 fps, and the vapor quality was 75% at 715°F. Exposure to these conditions produced no corrosion, mass transfer, or erosion in the blade section.

e. Condenser

Examination of the tapered tubes on the mercury side of the condenser indicated very little corrosion of tube walls. No mass-transfer deposits were found in the condenser tubes.

f. Mercury Pumps

(1) 9Cr-1Mo Steel Pump

This pump was operated for a total of 3971 hours in CL-3. It was then disassembled and inspected for wear, mass-transfer deposits, and erosion. The inspection indicated some Teflon-journal wear (Figure 25, Parts 6 and 12), a crack in the flange area of the front bearing housing (Figure 25, Part 4), and wear marks on the lower side of the hydraulic equalizing grooves. The journal wear amounted to approximately 0.025 in. The crack in the front bearing housing appears to have resulted from thermal stress in a weld area. Considerable weld metal was machined off during the finishing operation, and the indications are that the heat buildup in this area was caused by rubbing of the impeller's hydraulic balancing vanes against the housing.

There were also indications that the shaft was rubbing on the bottom of the shaft hole. The rub marks were all on the lower side of the case, indicating that the Teflon-journal wear was excessive and allowed the impeller shaft to drop and rub, thereby causing abnormal heat buildup. No mass-transfer deposits or erosion were found inside the impeller case or on the impeller (Figure 25, Parts 1 and 2).

(2) 405 SS Pump

This pump was operated for 421 hours in CL-3 as a standby pump. Its total operating time was 1494 hours, including operation in a pump-test loop and in CL-3, before disassembly. On visual inspection, the pump was found to be in satisfactory condition and reusable after the installation of new Teflon journals. No mass-transfer deposits were found on the impeller or impeller case.

g. Valves and Tubing

Three valves and a check valve in the all-liquid section of the mercury system were disassembled. The valve seats, valve parts, and tubing showed no indication of corrosion or mass-transfer buildup.

3. NaK-Condensing System

Examination of the NaK side of the mercury condenser, the NaK heat exchanger, and the tubing that made up the condensing system revealed no mass transfer or corrosion. No material-corrosion problem had been expected, in view of the low operating temperature of this system (700°F max).

4. Discussion

The evaluation of CL-3 to date indicates that the main corrosion and materials problem with the SNAP-8 system is likely to arise in the mercury side of the boiler. The solubility of 9Cr-1Mo steel in mercury at the expected boiler temperatures is such that corrosion will occur. The mercury-flow pattern during boiling apparently will control the location and severity of boiler-tubing corrosion. Exterior and interior cracking of the 9Cr-1Mo steel boiler tubing may also occur. The mechanism of this cracking has not been determined; because most of it occurred in the area of greatest heat transfer, it is probably associated with thermal stress or thermal fatigue. It may, however, only be common to the undesirable operating state of the CL-3 boiler.

Other areas of the mercury system appear to be free of serious corrosion and mass-transfer problems when 9Cr-1Mo steel is used. Essentially no mass-transfer deposits were found in the condenser and the liquid lines in the loop. The corrosion products generated in these areas apparently remained suspended in the mercury and/or floating at the mercury interfaces, eliminating the problem of tube restriction found in many liquid-metal systems.

B. CORROSION LOOP 4

1. Operation

a. NaK System

Assembly of the NaK primary and condensing loops was completed, and the loops were checked out. The primary loop was started on 12 January 1965, and the condensing loop on 14 January. Checkout and startup procedures were accomplished in accordance with Ref. 18. Both loops performed satisfactorily during the start and throughout this report period. The oxide control of the primary loop was as follows:

| Date | Accumulated Operating Time hours | Oxide Level, ppm | Remarks |
|---|---|---|---|
| 1/14/65 | 24 | 32 | Cold-trapped 6 hours (trap temperature 225°F), oxide level reduced to 25 ppm |
| 1/27/65 | 40 | 25 | Prior to mercury injection |
| 3/9/65 | 660 | 24 | After completion of Run 1 |
| 5/17/65 | 1100 | 18 | -- |

b. Mercury System

The mercury system was assembled and checked out. Prior to installation of the system into the loop, the boiler was flushed in Freon (see Ref. 15, p. 30). Table 2 provides information on the operation of CL-4 during this report period. Mercury boiling was started on 28 January 1965 and continued through 24 February. Figure 26 shows the boiler performance during this period.

Superheated vapor was obtained immediately when mercury was injected into the boiler, but the rated design conditions were not attained. At the start, the mercury flow rate was approximately 250 lb/hour; the boiler-outlet pressure was 80 psia and the boiler-outlet temperature 1100°F. Mercury boiling was continued until 9 February, when the rated boiler-outlet pressure was attained (265 psia). The loop was operated continuously until 12 February when an electric-power outage in the area caused a shutdown for 2 hours. Upon restarting, the boiler performance did not recover until after 48 hours of operation. The system operated until 24 February when a leak in the cooling-water piping caused a shutdown. A total of 650 hours of operation was accumulated.

A slight improvement in boiler performance was noted, on the basis of NaK-shell-temperature profiles taken at the beginning and end of the run (Figure 26). These profiles showed that some conditioning had occurred in the last 15 ft of the boiler; they also indicated that an extended run-in period is required to condition the boiler.

A modified mercury-inlet-plug insert was installed in the boiler. It has two wire-wrapped sections of different pitch - 1/8 in. in the mercury-preheat section and 3/4 in. in the succeeding (low-quality) section. The geometry of, and flow dynamic values for, the preheating regions of the original and the modified plug inserts are summarized below.

| | Original Inlet Plug (No. 1) | Modified Inlet Plug (No. 2) |
|---|---|---|
| Tube ID, in. (nominal) | 0.397 | 0.397 |
| Plug-insert diameter, in. | 0.301 | 0.301 |
| Wire diameter, in. | 0.049 | 0.049 |
| Wire pitch, in. | 3/4 | 1/8 |
| Flow-channel cross section, $ft^2$ | $20.1 \times 10^{-5}$ | $2.94 \times 10^{-5}$ |
| Hg flow rate, lb/hour (nominal) | 500 | 500 |
| Liquid density, $lb/ft^3$ | 760 | 760 |
| Liquid-Hg helical velocity, fps | 0.91 | 6.22 |

A test run with the modified plug showed an immediate heat-transfer improvement in the inlet-plug region and completion of mercury boiling at a length of about 30 ft (compare Curves 1 and 2, Figure 27). Considerable conditioning was noted during the subsequent 16 hours of operation (Curve 3, Figure 27). The boiler was then shut down and restarted to confirm the conditioning phenomena. On the restart, it exhibited the same heat transfer and stable boiling performance as before (the range of pressure fluctuation was 0.4% of the boiler-outlet pressure, 225 psia).

The boiler was then restarted with the original inlet plug (3/4-in. pitch, 60-in. length); its performance was good, indicating that the boiler-tube surface had been conditioned. Although the data provided only qualitative information, the multipitch plug demonstrated improved boiler performance, and a boiler-inlet-plug test program was initiated.

### 2. Testing of Boiler-Inlet Plugs

#### a. Instrumented Plug

Four different instrumented plugs (Figure 28), were tested for design evaluation. They were provided with pressure taps downstream of the tight-pitch region and at the end of the plug. The liquid-mercury and two-phase pressure-drop factor and heat-transfer information for the plugs were established on the basis of pressure readings from these two taps, the boiler inlet and outlet pressures, and the NaK-shell temperature. The pressure sensors for these taps and the mercury-boiler inlet and outlet were strain-gage-type pressure transducers. An electronic Visicorder was used for pressure readout. The mercury-outlet temperature was sensed with an immersion-type thermocouple, and the NaK shell temperatures with skin-type thermocouples. Figure 29 presents a typical plot for an instrumented-inlet-plug data point.

#### b. Test-Run Summary

In addition to the testing of the four different instrumented plugs, a run was also made without a plug insert. Each of these runs included

(1) Boiling tests to determine the effect of operating-parameter changes on boiler performance (operating variables: NaK flow, NaK-boiler-inlet temperature, and mercury-boiler-outlet pressure)

(2) Isothermal liquid-mercury flow tests to determine the pressure drop across the tight-pitch and loose-pitch regions of the inlet plug.

Table 2 summarizes the results of these runs and describes the plugs. Detailed data are provided in Refs. 19, 20, and 21 and were forwarded to the SNAP-8 Heat Exchanger Group for analysis.

The corrosion patterns in the various boiler-inlet plugs (Table 2) indicate that the increased liquid velocity and improved boiler performance caused noticeable corrosion in plug regions with operating times as short as 100 hours. In addition, the difference in the corrosion patterns for Runs 1 and 4 indicates the influence of boiler performance on the corrosion pattern found in the boiler-inlet region.

In these tests the clearance between the inner surface of the boiler tubing and the outside of the plug was found to be critical. If this clearance is not controlled, the differential pressure and liquid velocity in the tight-pitch region of the plug will be reduced because of bypass flow.

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TABLE 1

CHROMATOGRAPHIC ANALYSIS OF MIX-4P3E

| | Composition, wt% | | | | | |
|---|---|---|---|---|---|---|
| | Low Boilers* | Isomers** | | | | |
| | | o-o | m-o | o-p + m-m | m-p | p-p |
| Shell Development Company certification | 0.4 | None | 5.2 | 52.6 | 39.2 | 2.6 |
| Von Karman Center check analysis (av. of 14 specimens) | 0.49 | 0.13 | 5.2 | 52.8 | 38.6 | 3.5 |
| Decanted fluid after crystallation | 0.5 | 0.2 | 5.3 | 51.3 | 40.3 | 2.4 |
| Crystalline material*** | 0.5 | 0.2 | 5.2 | 50.7 | 38.9 | 4.5 |

*Low boilers = organic substances in mix-4P3E that boil at a lower temperature than the mix-4P3E; among them are phenol, phenoxyphenol, and isomers of diphenoxybenzene.

**Isomer definitions:

o-o = ortho-ortho phenoxyphenyl ether
m-o = meta-ortho phenoxyphenyl ether
o-p = ortho-para phenoxyphenyl ether
m-m = meta-meta phenoxyphenyl ether
m-p = meta-para phenoxyphenyl ether
p-p = para-para phenoxyphenyl ether.

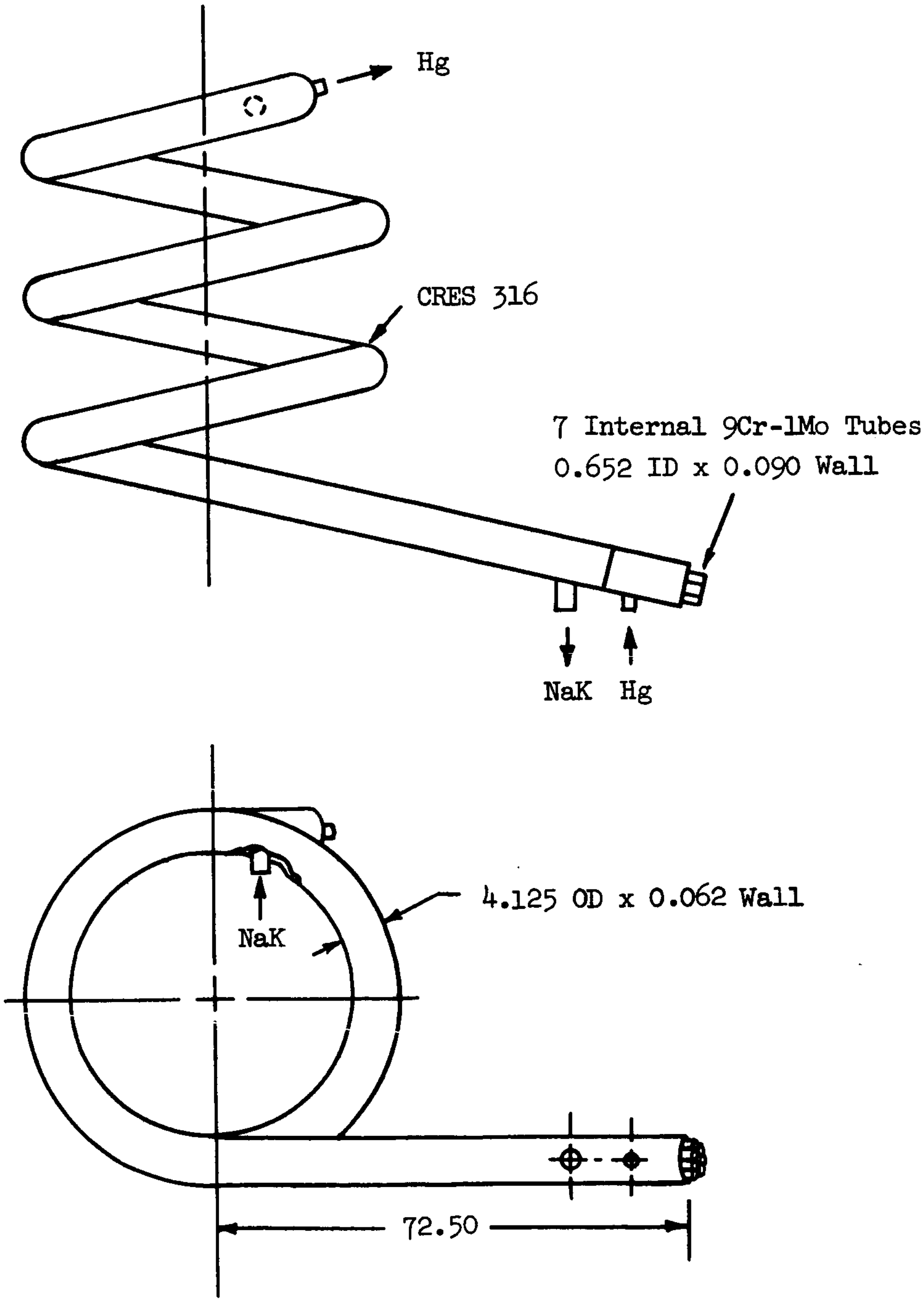
***Additional tests of this material are being conducted to ensure that incomplete removal of mix-4P3E fluid from the precipitate did not mask the true composition of the solid.

TABLE 2

OPERATING INFORMATION, CORROSION LOOP 4

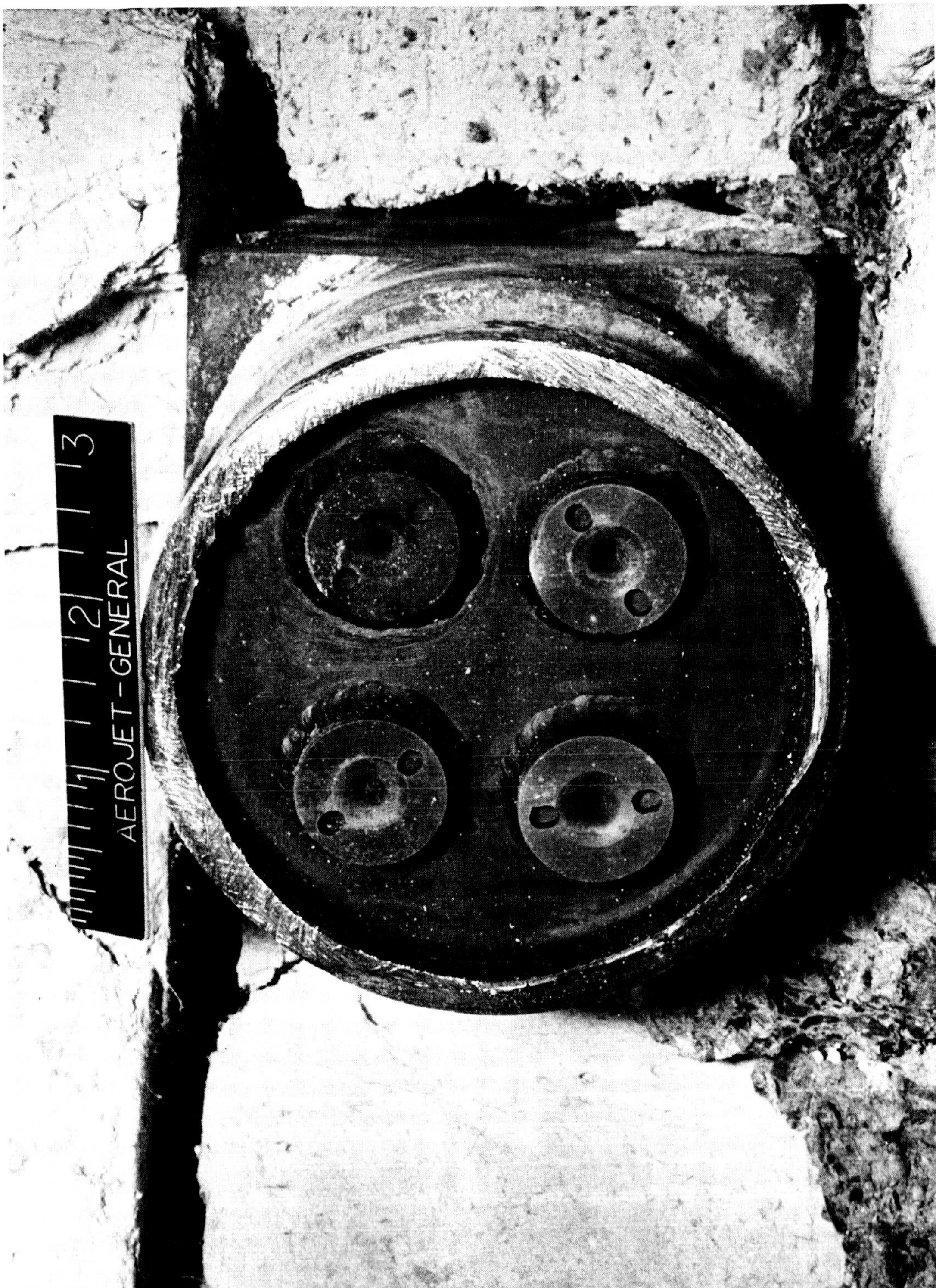
| Run No. | Test Period (1965) | Inlet-Plug Description* No. | Dimensions, in. Length | Dimensions, in. Pitch | Type of Helix | Operating Time, hours (Hg Boiling) | No. of Data Points | Remarks |
|---|---|---|---|---|---|---|---|---|
| 1 | 28 Jan-24 Feb | 1 | 60 | 3/4 | W | 650 | - | Start-up and corrosion testing. No corrosion was apparent when the plug was removed. |
| 2 | 10 Mar-12 Mar | 2 | 18<br>36 | 1/8<br>3/4 | W<br>W | 52 | - | First test of multipitch inlet plug. On removal, corrosion was observed in the 1/8-in.-pitch section (on the wire and plug), and none was observed in the 3/4-in.-pitch section. |
| 3 | 17 Mar-19 Mar | 2 | Same as Plug 2 above | | | 52 | - | Additional tests with Plug 2 to confirm the results of Run 2. |
| 4 | 30 Mar-3 Apr | 1 | Same as Plug 1 above | | | 100 | - | Retest of Plug 1 from Run 1. Corrosion was observed throughout its length, and the exit end was mercury-wetted. |
| 5 | 8 Apr-12 Apr | 3 | 15<br>36 | 1/8<br>3/4 | M<br>W | 96 | 21 | First test of instrumented inlet plug. Corrosion was observed in the 1/8-in.-pitch section, as well as mercury wetting. Very little corrosion was noted in the 3/4-in.-pitch section. |
| 6 | 28 Apr-30 Apr | 4 | 15<br>36 | 1/8<br>1-1/2 | M<br>W | 77 | 19 | A slight amount of corrosion was observed on the machined threads. Their outside surfaces were scored, indicating that a close fit had obtained between the inner surface of the boiler tubing and the exterior of the plug. |
| 7 | 6 May-7 May | 4 | Same as Plug 4 above | | | 24 | 6 | Retest of Plug 4, which was removed from the boiler and reinstalled between Runs 6 and 7. |
| 8 | 12 May-19 May | 3a | 15<br>36 | 1/8<br>1-1/2 | W<br>W | 86 | 16 | Corrosion was observed in the 1/8-in.-pitch section, immediately following which the 1-1/2-in.-pitch section was mercury-wetted. |
| 9 | 26 May | - | - | - | - | 5 | 6 | No inlet plug was used. The boiler inlet and outlet pressures fluctuated widely (maximum at outlet ±45 psi). |
| 10 | 1 Jun-4 Jun | 3b | 15<br>24 | 0.170<br>1-1/2 | W<br>W | 72 | 8 | The 0.170- and 1-1/2-in.-pitch sections were mercury-wetted. |

*The plug covered the inlet portion of the mercury-boiler tube, where the liquid mercury was preheated and the initial, low-quality vapor was produced. The dimensions given represent the length of the wire-wrapped (W) or machined section and the pitch of the machined thread (M) or the wire wrapping (0.049-in.-dia wire). The first in a pair of dimensions (i.e., for Plugs 2 through 4) represent the initial portion of a multipitch plug.




Preliminary Reference Configuration
Tube-in-Tube Boiler (RPL-2), 30-ft Total Length

Figure 1

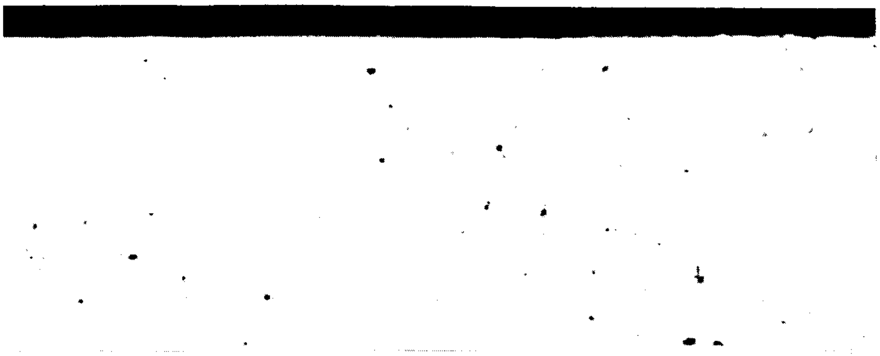



365-786

SNAP-8 -1 Boiler, Mercury-Inlet Manifold Showing Blocked Inlet Restrictor (Upper Left)

Figure 2

304 SS

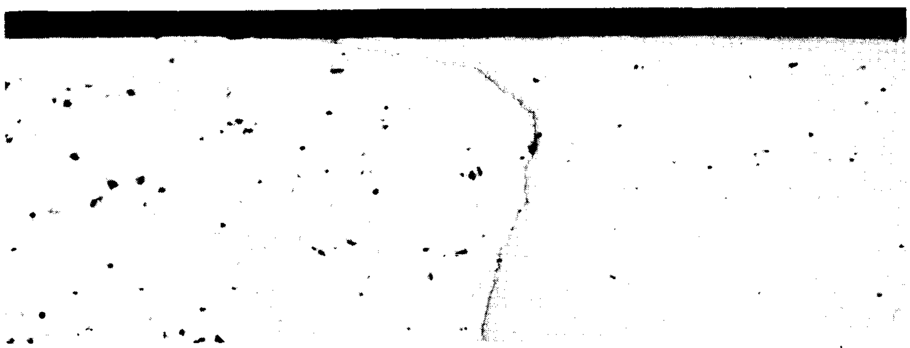


Al

As Polished L-8731 150X

a. Unexposed Specimen

Al

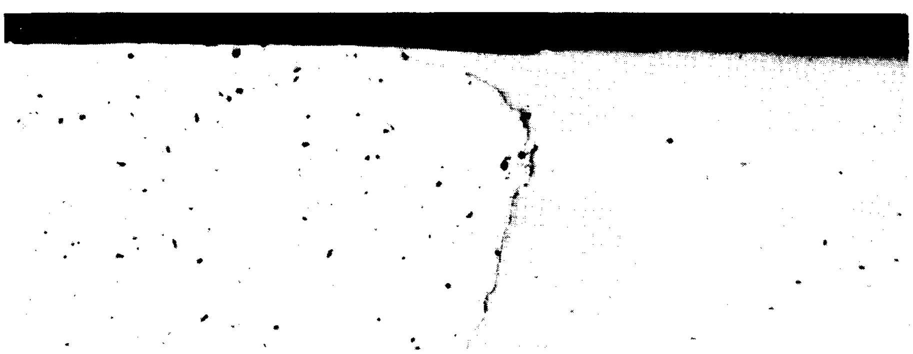


304 SS

As Polished L-8729 150X

b. Specimen Exposed to Mix-4P3E

Al

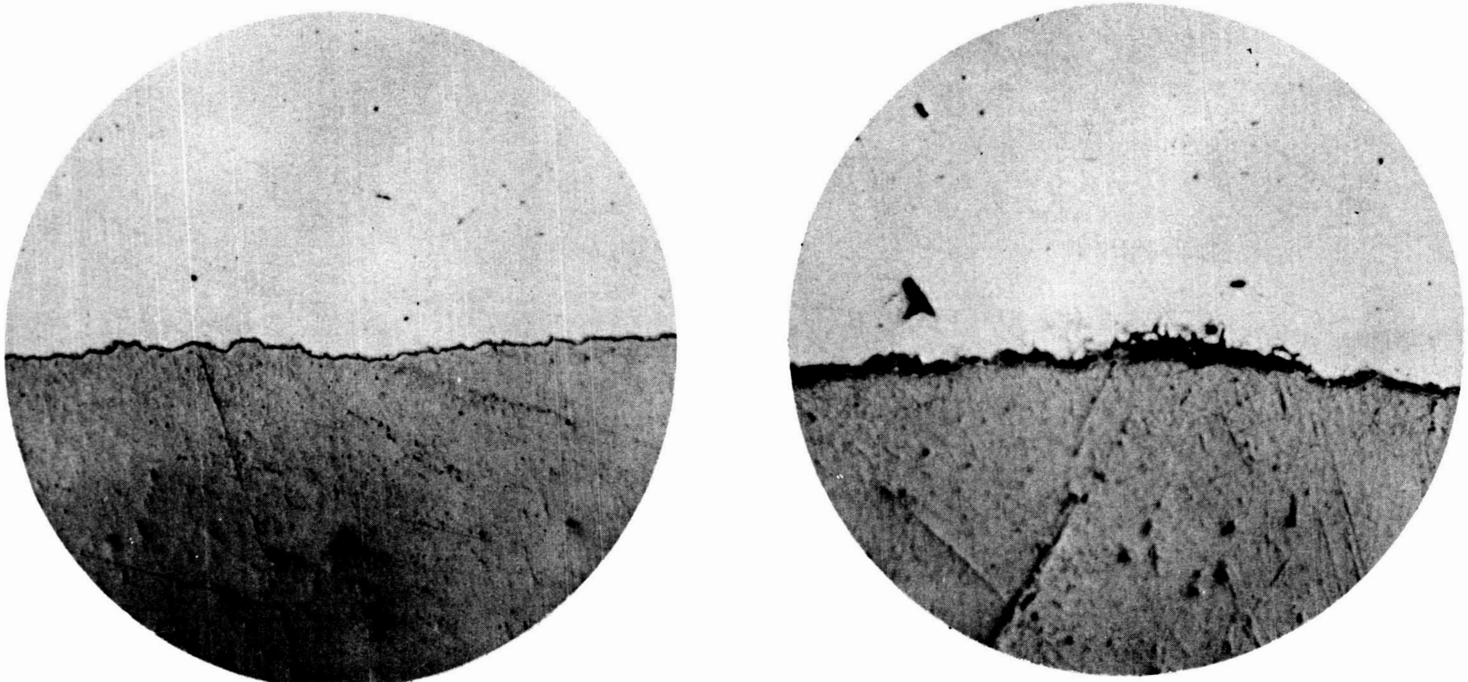


304 SS

As Polished L-8730 150X

c. Specimen Exposed to Mix-4P3E + 1 vol% $H_2O$

Aluminum/304 SS Hot-Coextruded Transition Joint After Exposure to Mix-4P3E Solutions for 2500 Hours at 250°F in Evacuated Glass Capsule (Surface Exposed to Liquid Appears at Top)



AS POLISHED NMI-4a 500X

AFTER BENDING

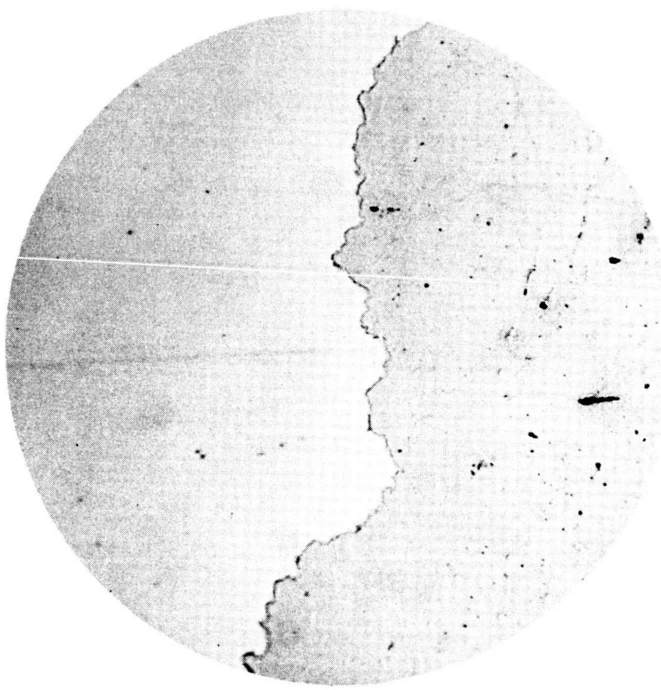
AS POLISHED NMI-6c 500X

AFTER BENDING AND THERMAL CYCLING

0.684-IN.-ID TUBING

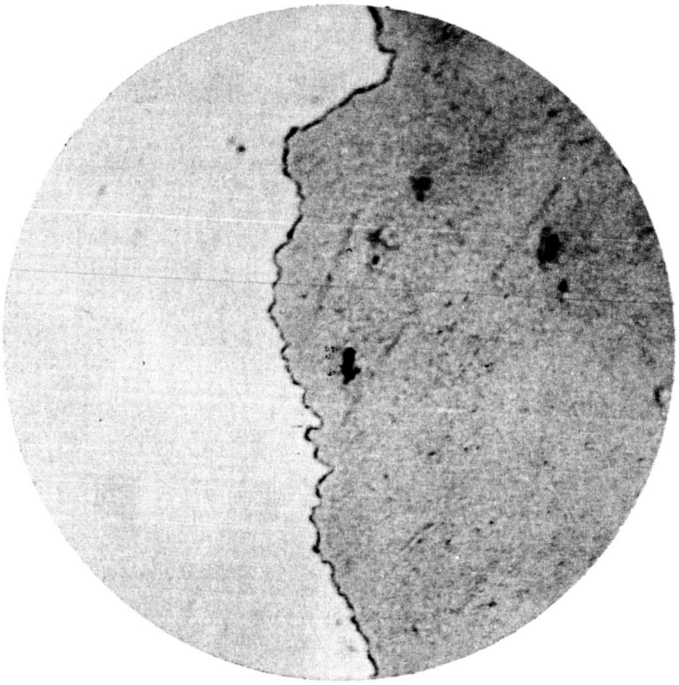
Interface of Columbium/316 SS Tubing (0.684-in. ID)
After Bend Test and Thermal Cycling (Cb at Bottom)

Figure 4



**AS POLISHED NMI-2 500X**

**AFTER BENDING**



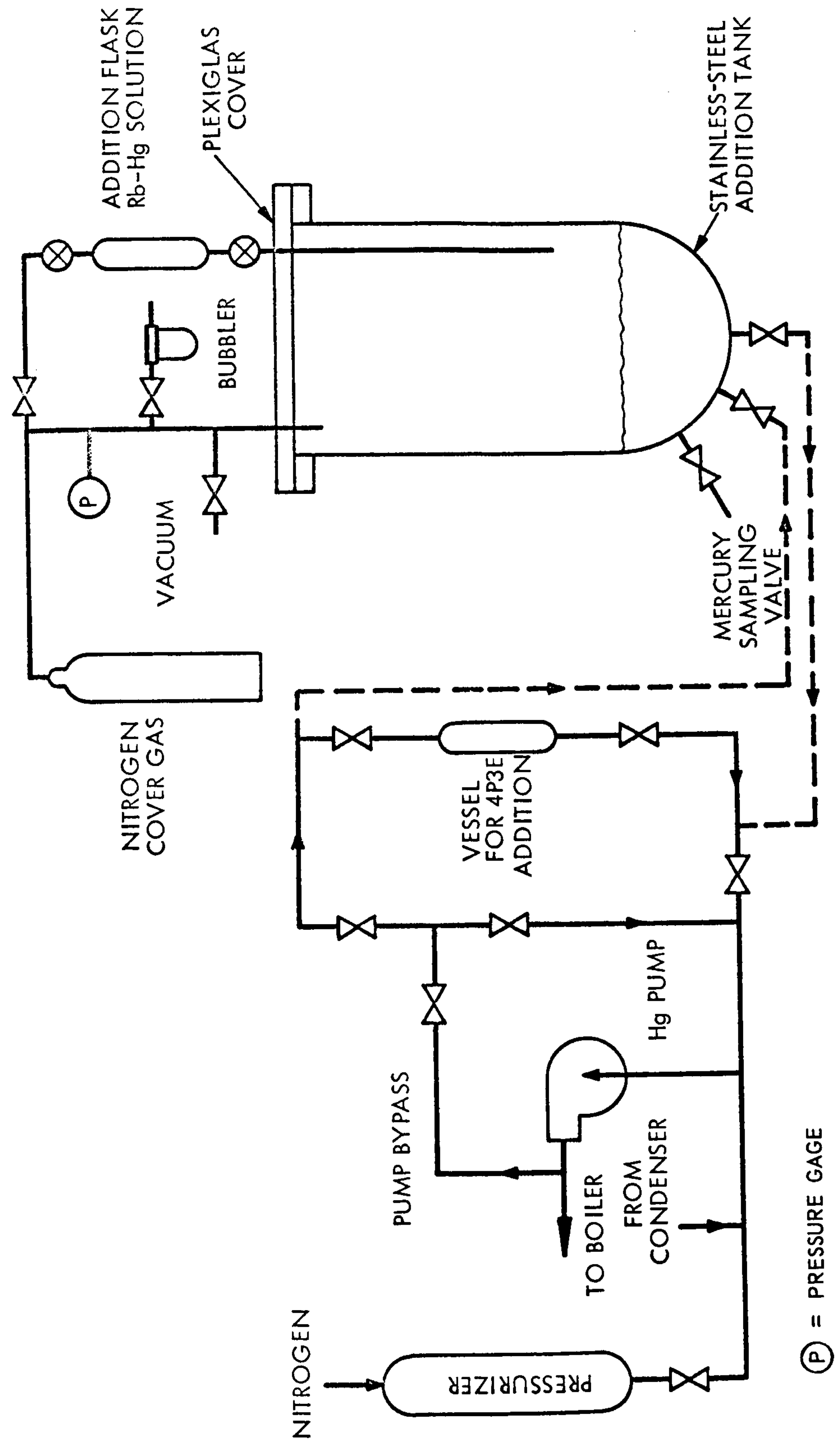
**AS POLISHED NMI-3 500X**

**AFTER BENDING AND THERMAL CYCLING**

**0.400-IN.-ID TUBING**

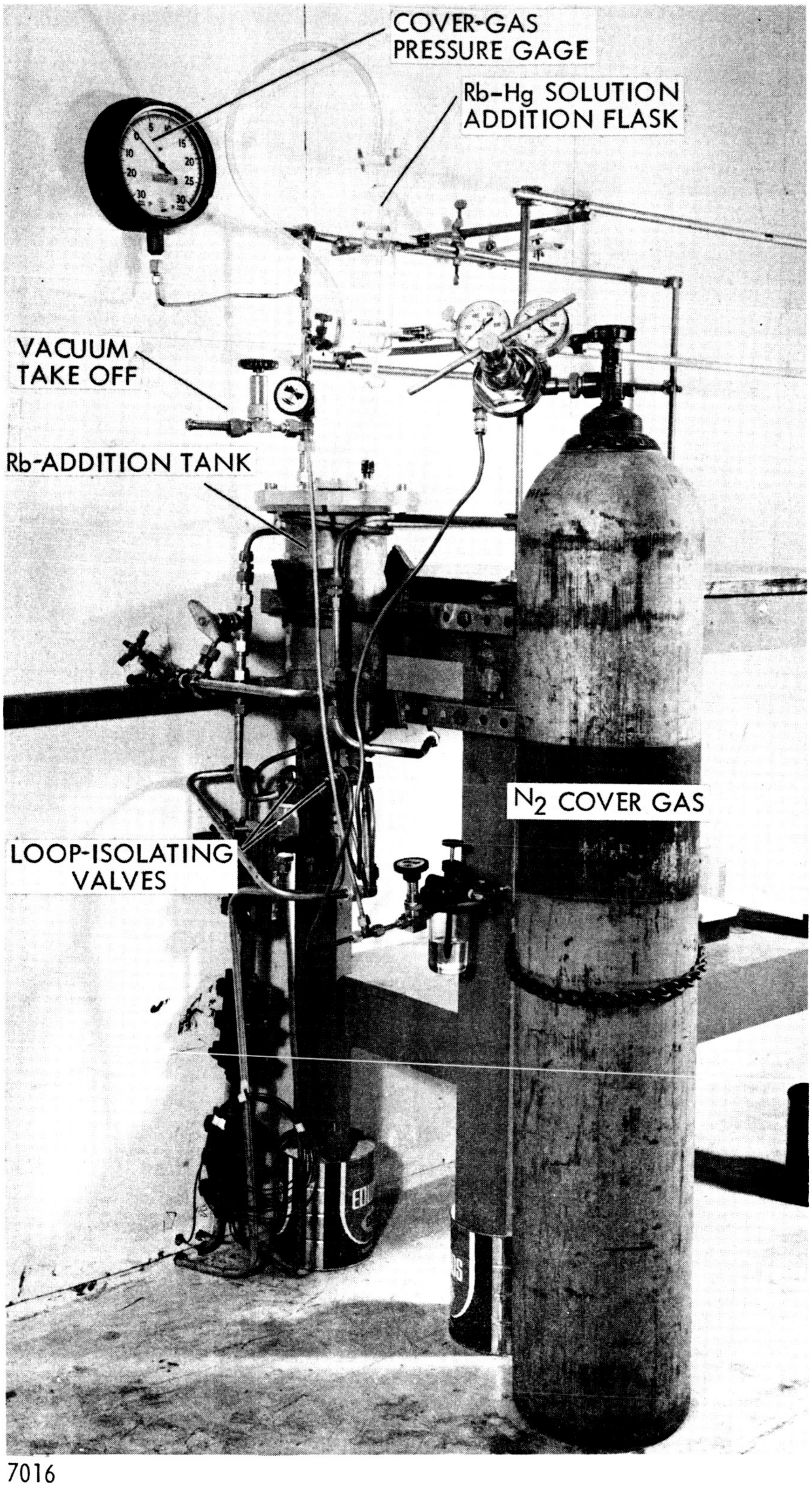
Interface of Cb/316 SS Tubing (0.400-in. ID)
After Bend Test and Thermal Cycling (Cb at Bottom)

Figure 5



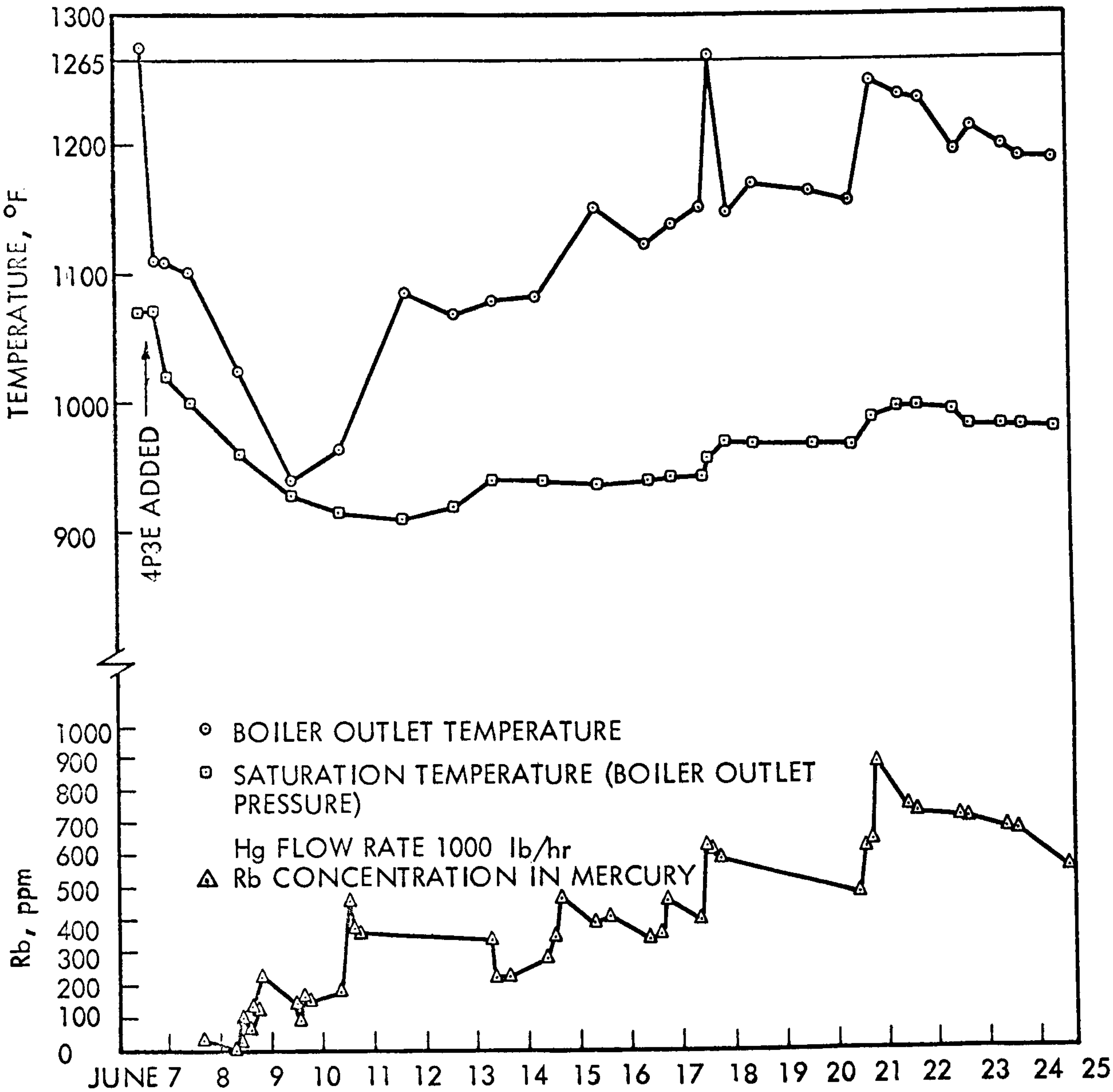

Rubidium and Mix-4P3E Addition System (Schematic), Component Test Loop 2 (CTL-2)

Figure 6



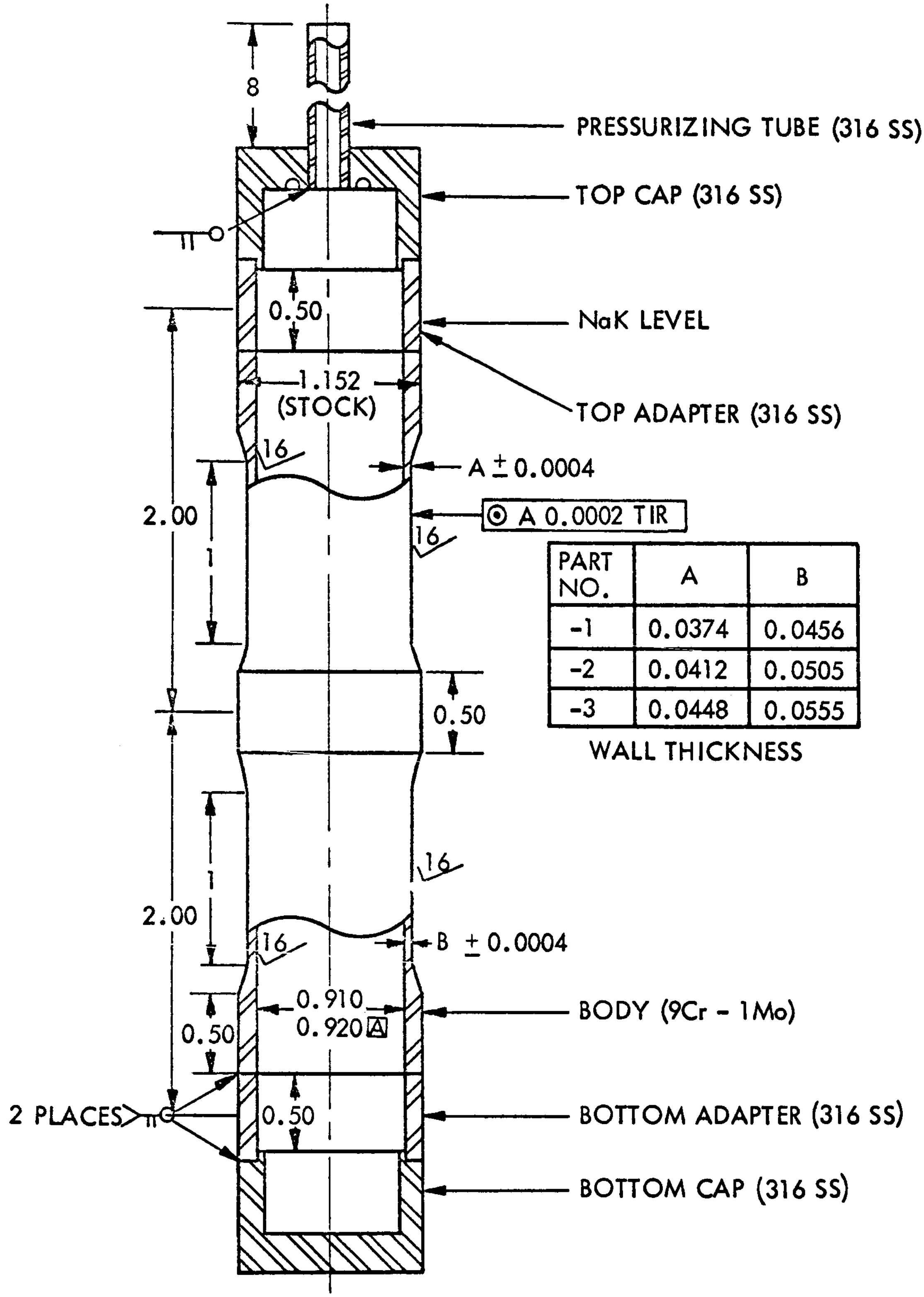

Rb-Addition System, CTL-2

Figure 7




Boiler Performance After Mix-4P3E and Rb Additions, CTL-2

Figure 8

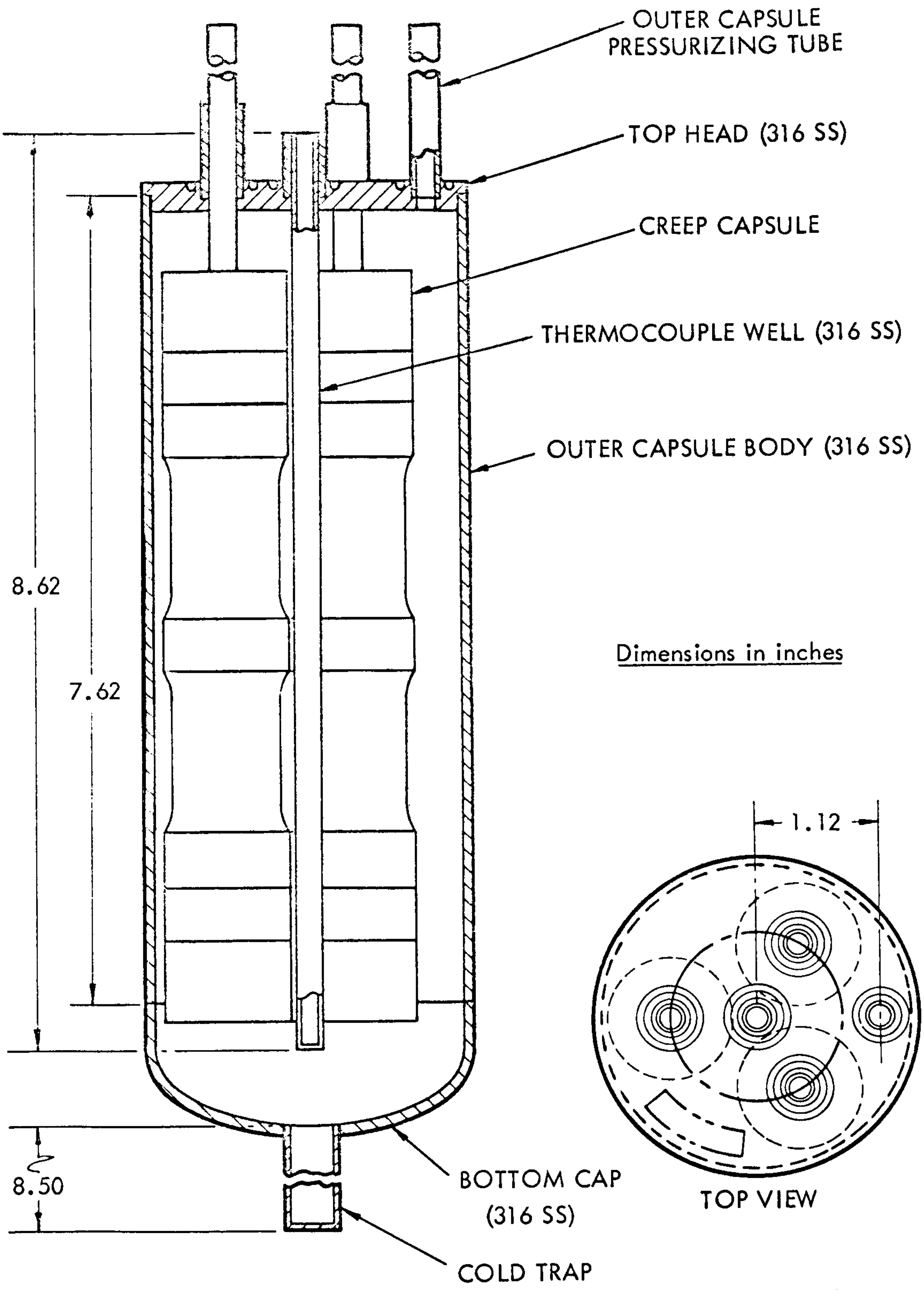



| PART NO. | A | B |
|---|---|---|
| -1 | 0.0374 | 0.0456 |
| -2 | 0.0412 | 0.0505 |
| -3 | 0.0448 | 0.0555 |

WALL THICKNESS

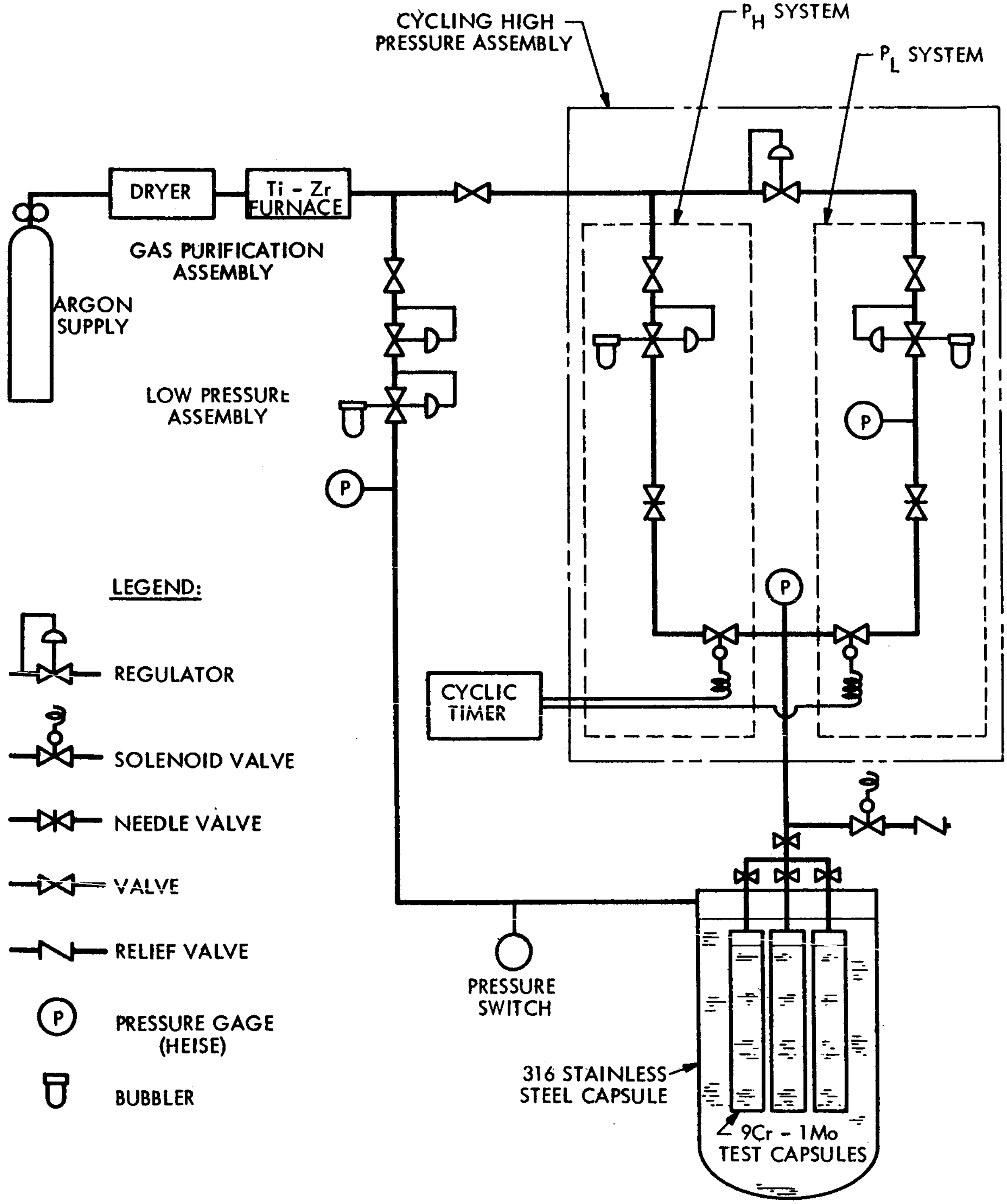
Creep Capsule

Figure 9



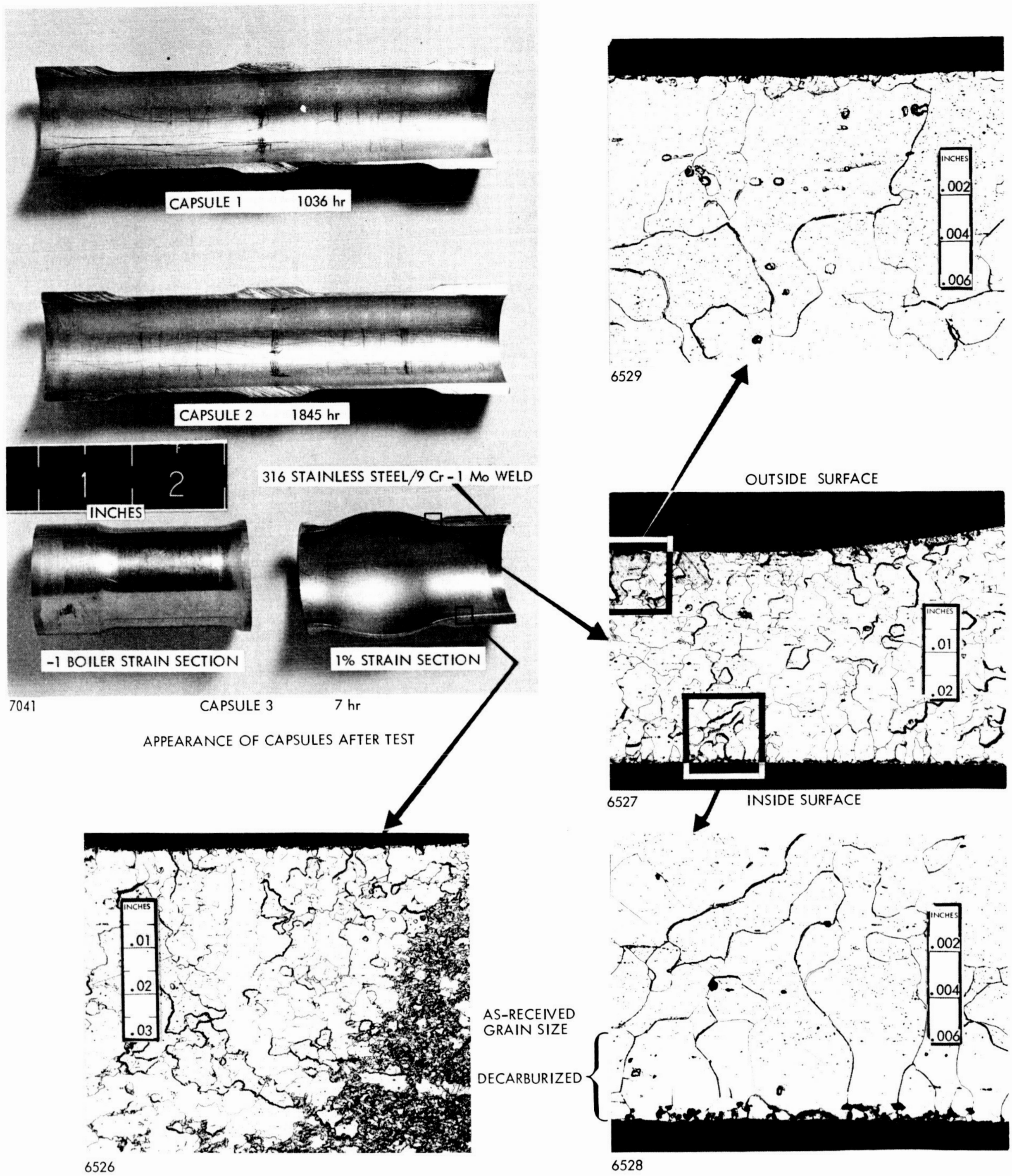

Outer Capsule Assembly

Figure 10



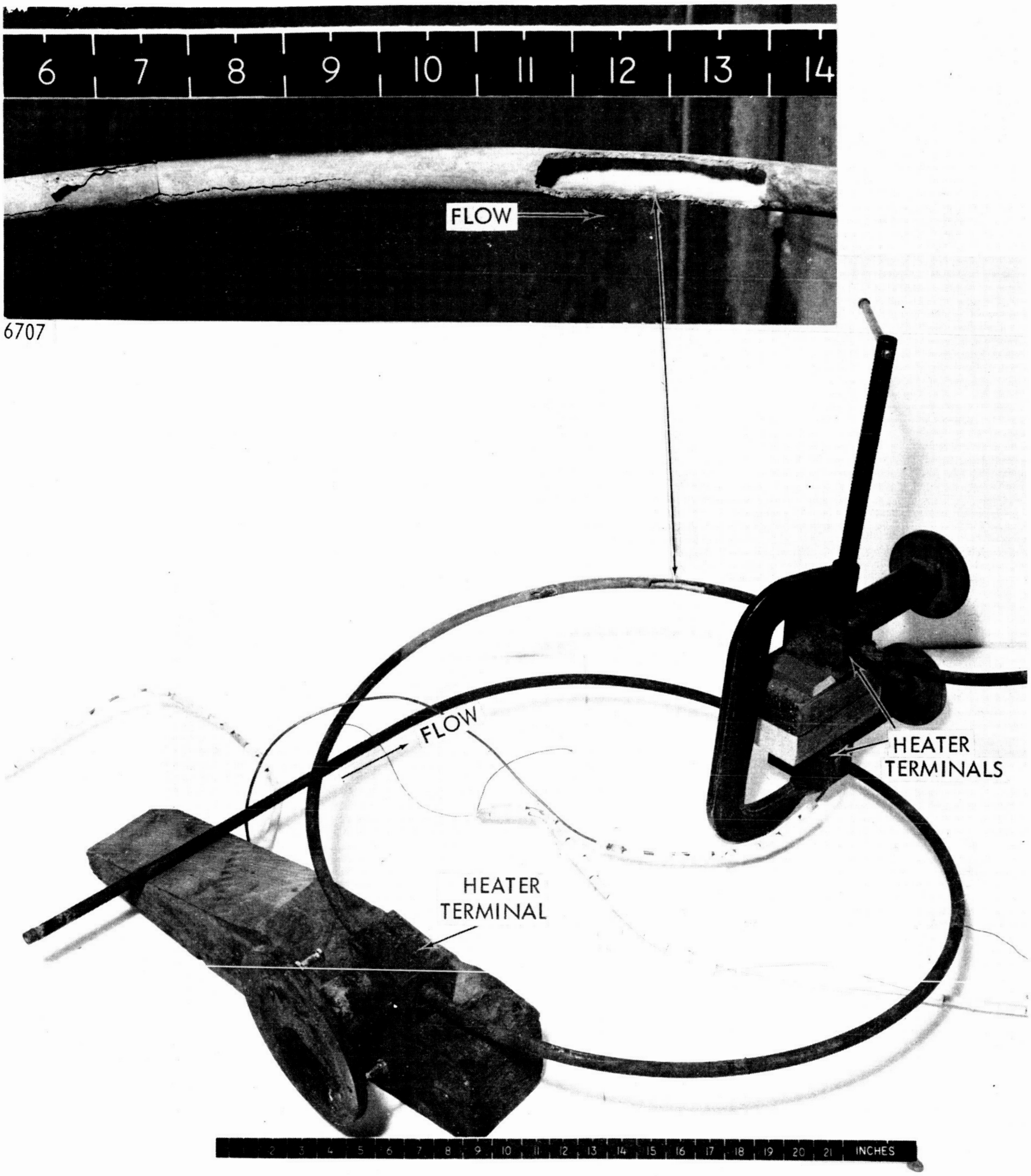

Gas-Pressurization System

Figure 11




Results from Cyclic-Creep Test (Vilella's Etch)

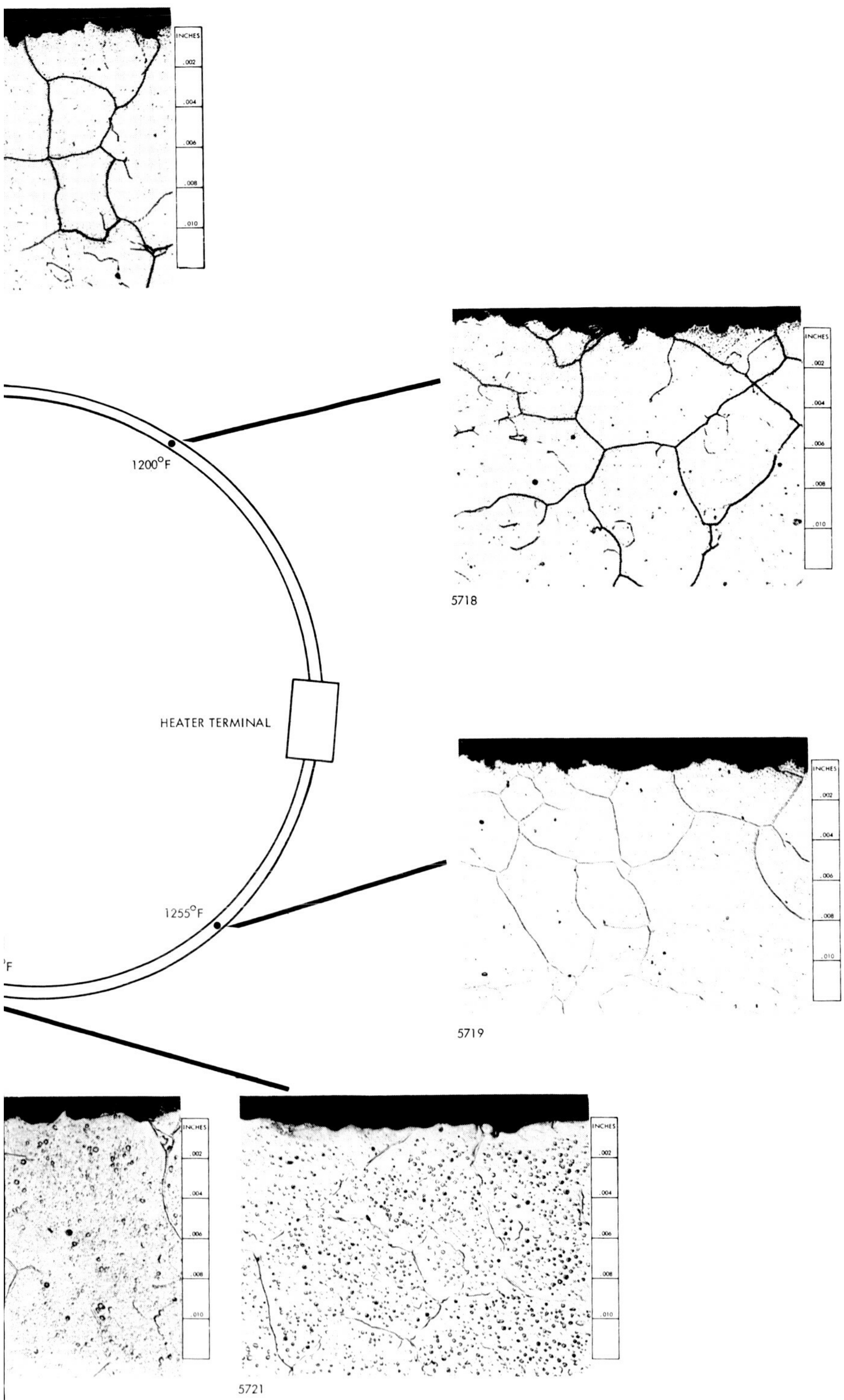
Figure 12




6707

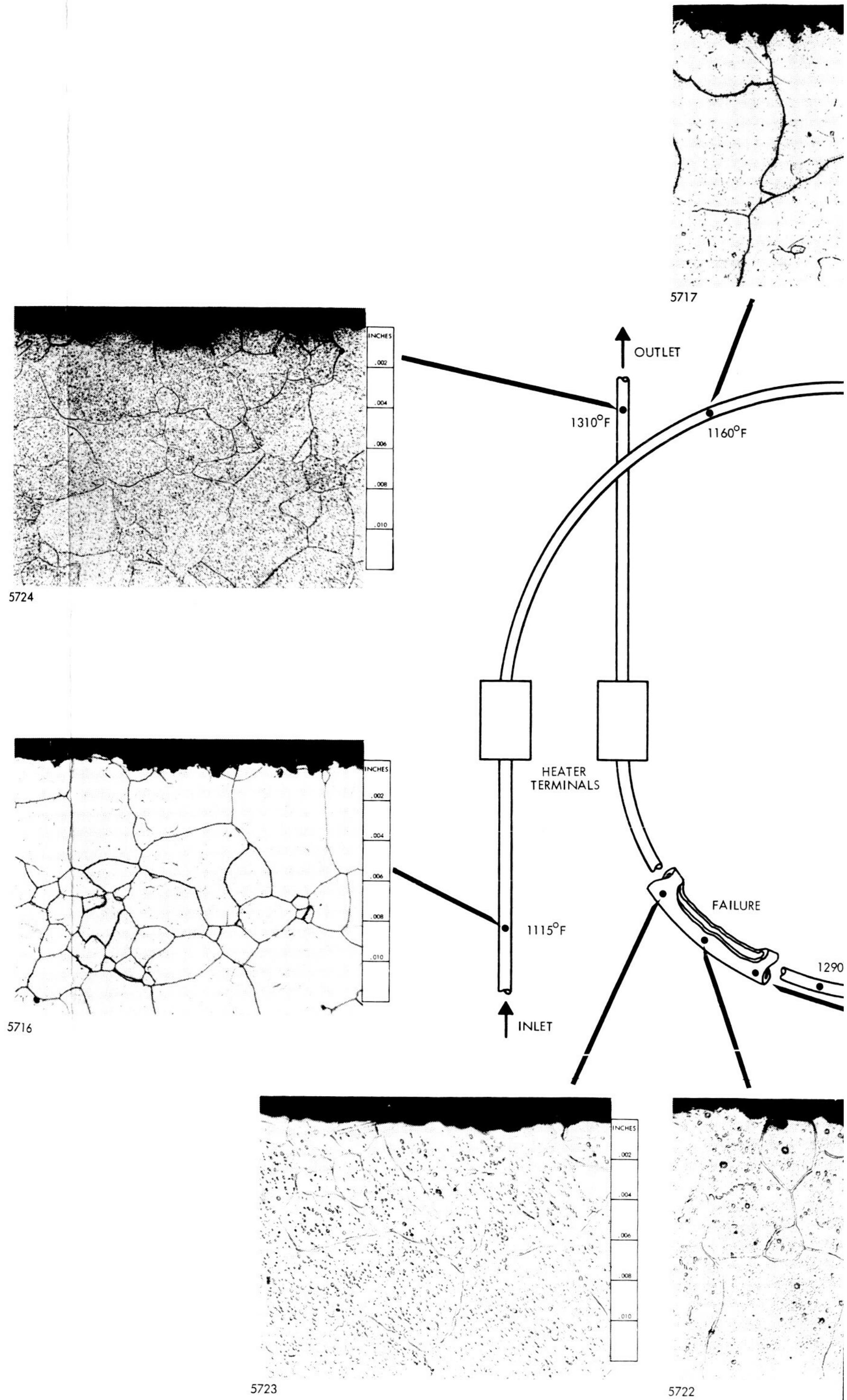
6727

NaK-Heater Tube Showing Area of Failure, Corrosion Loop 3 (CL-3)




er Tubing, CL-3 (Vilella's Etch)

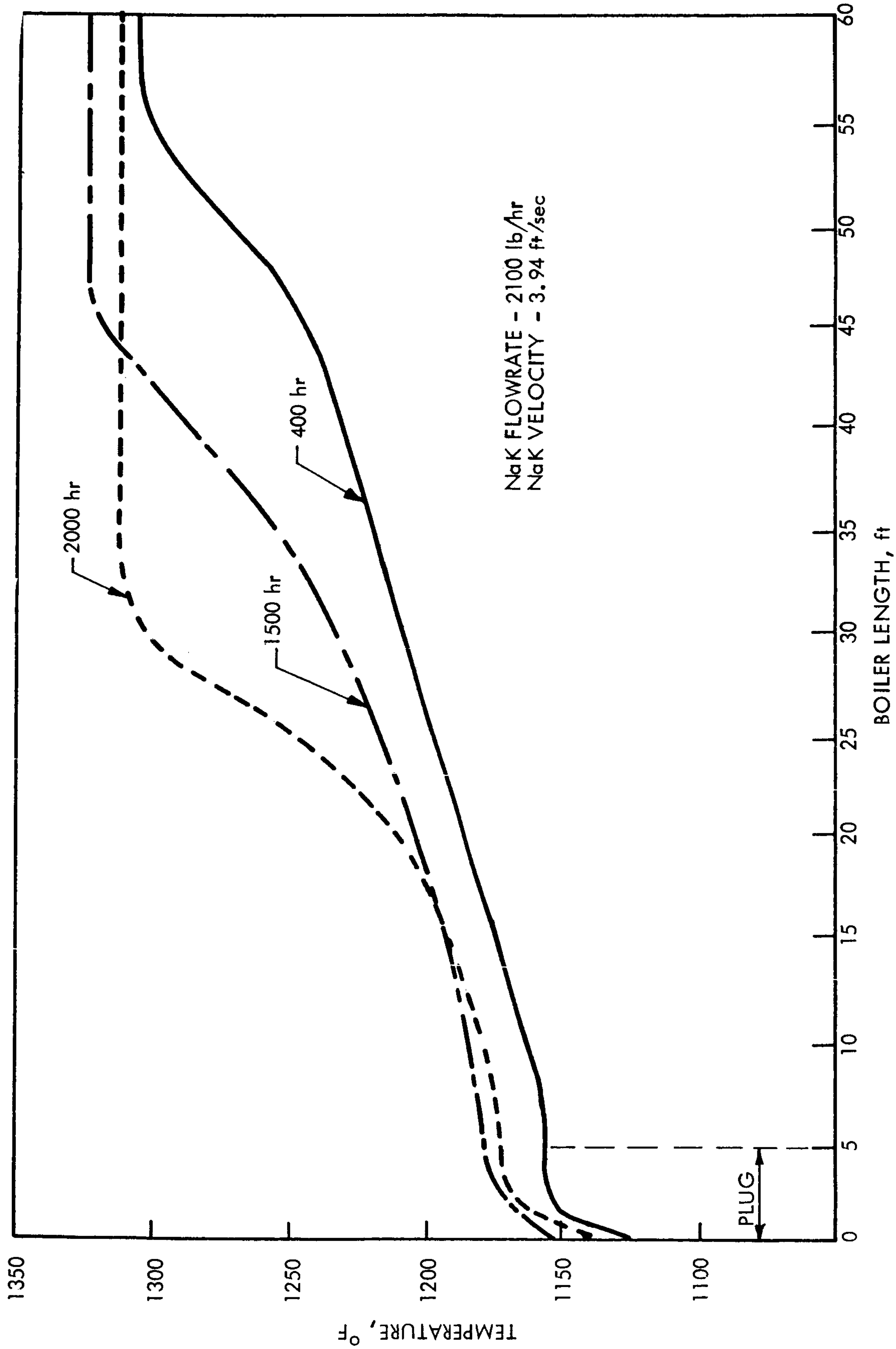
19②




Microstructure of 316 SS NaK-Heat

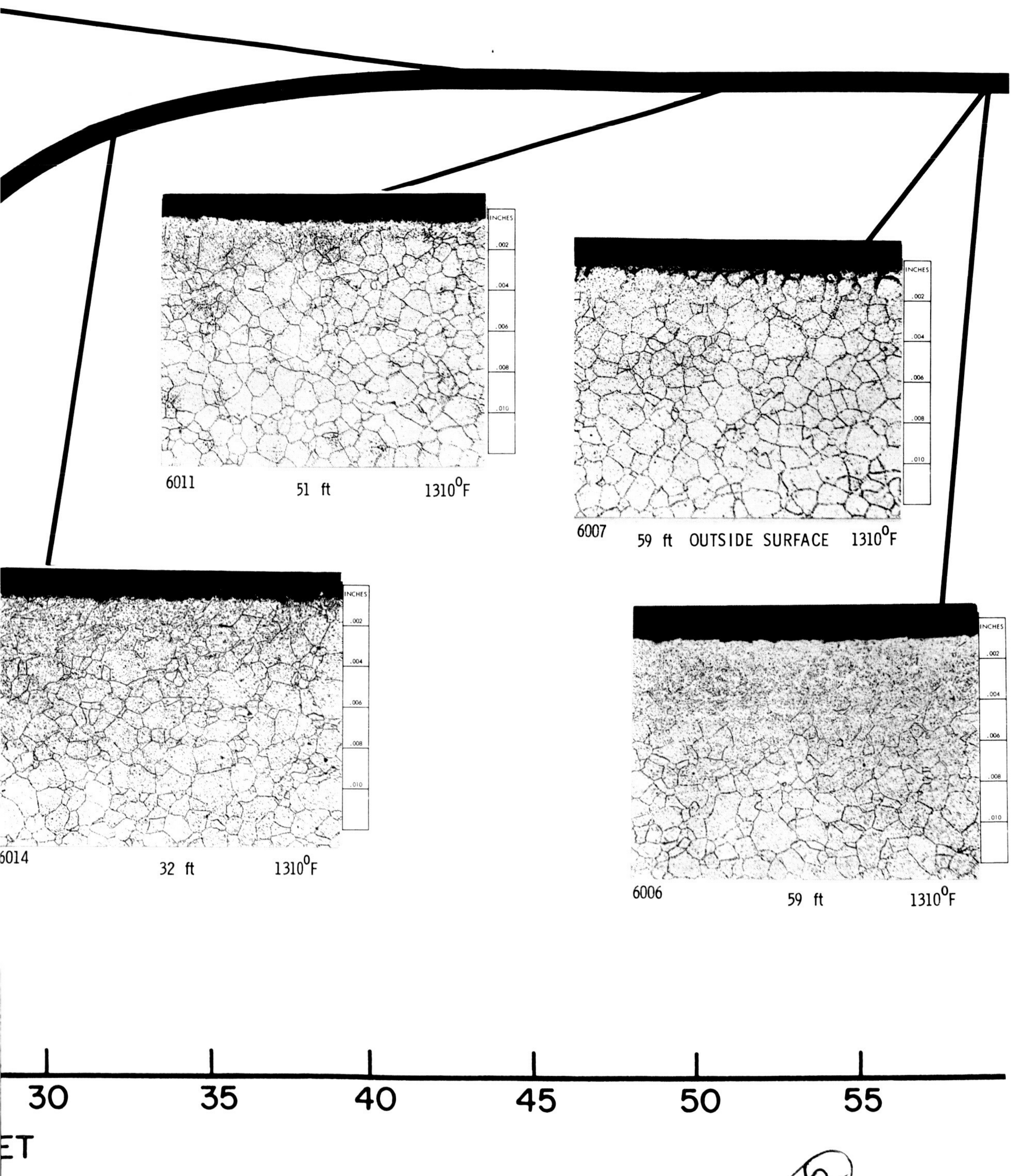
(1)

Figure 14



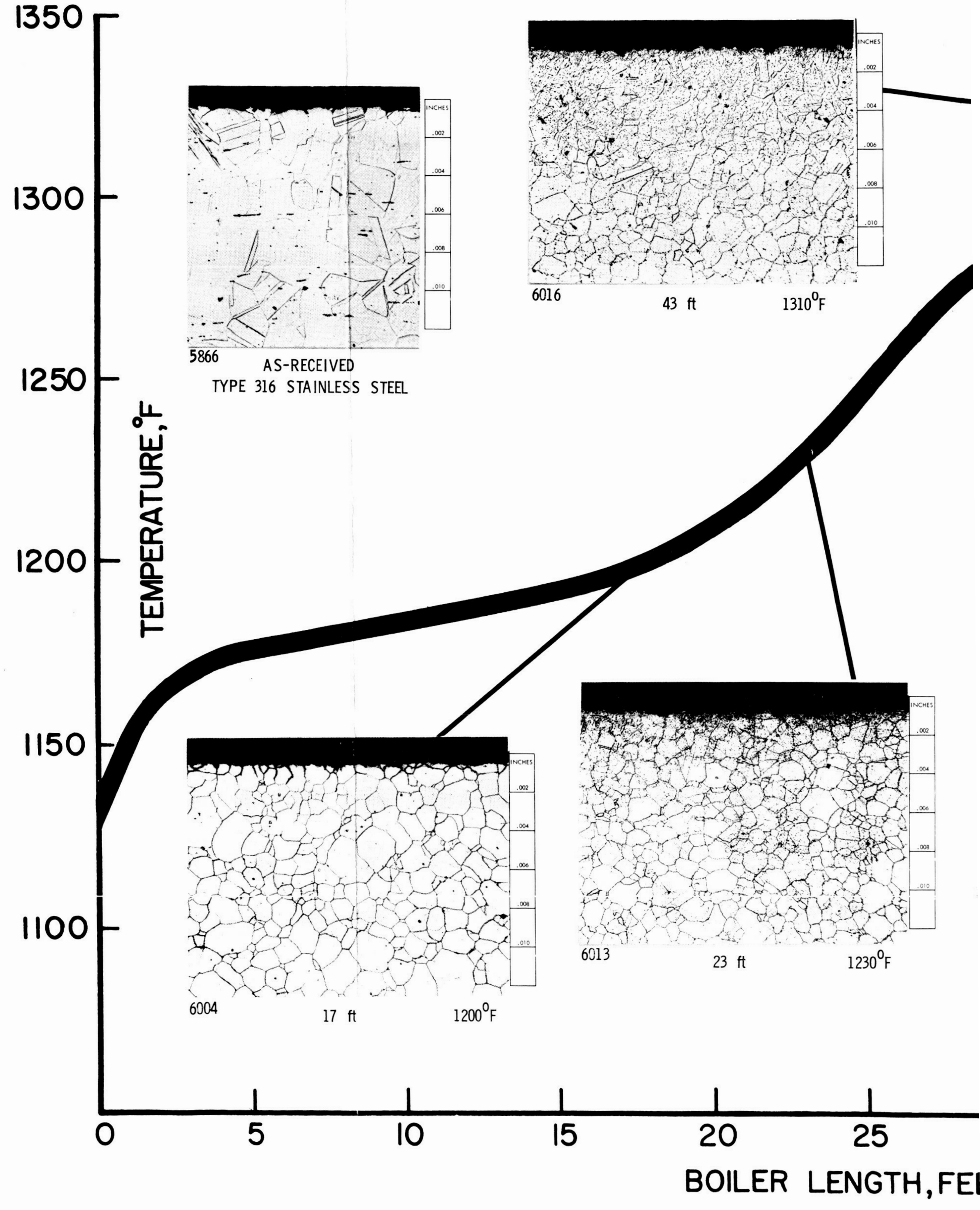

NaK-Temperature Profiles in Mercury Boiler, CL-3

Figure 15



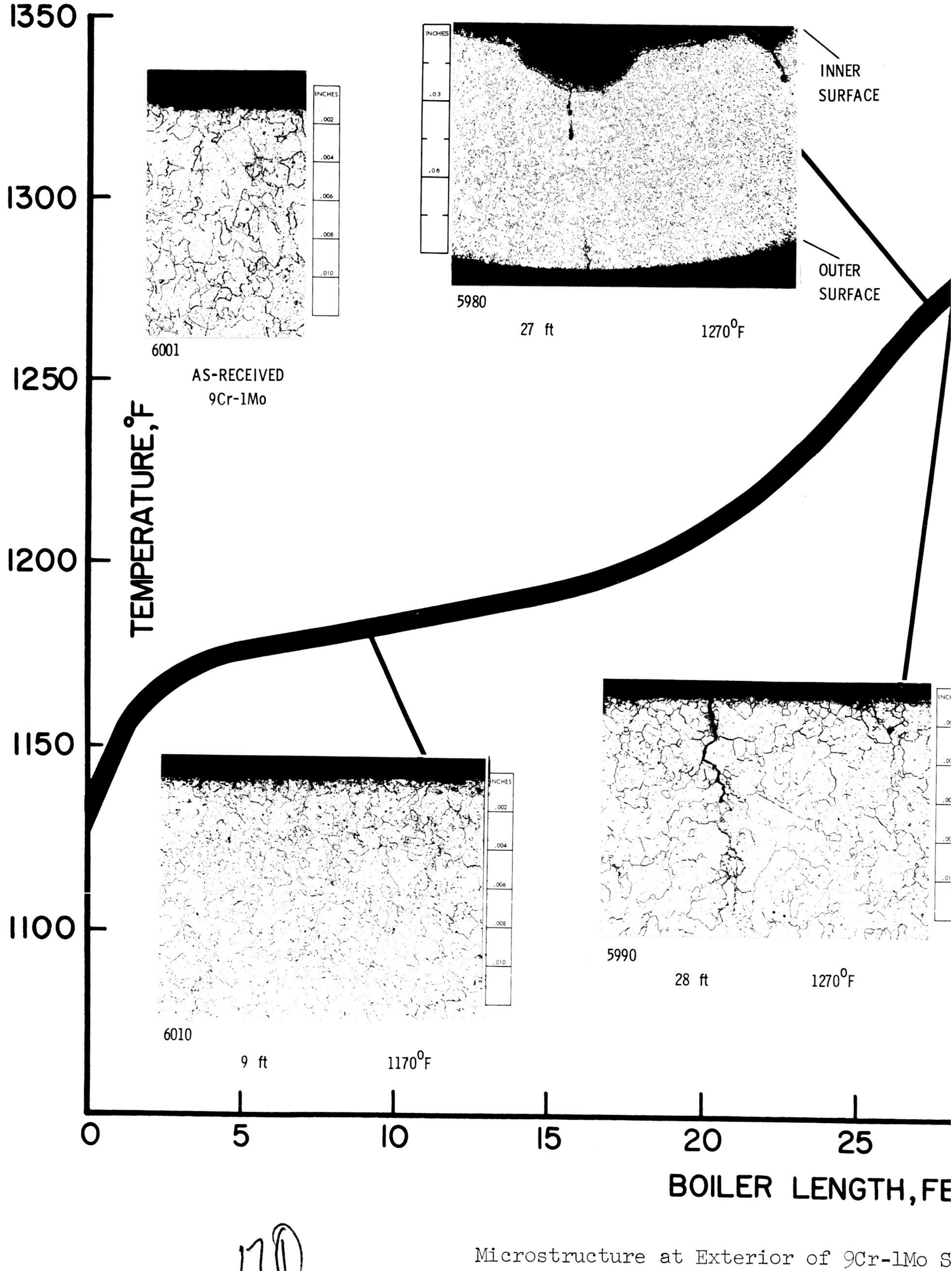

ell, CL-3 Boiler (Vilella's Etch)

16-2



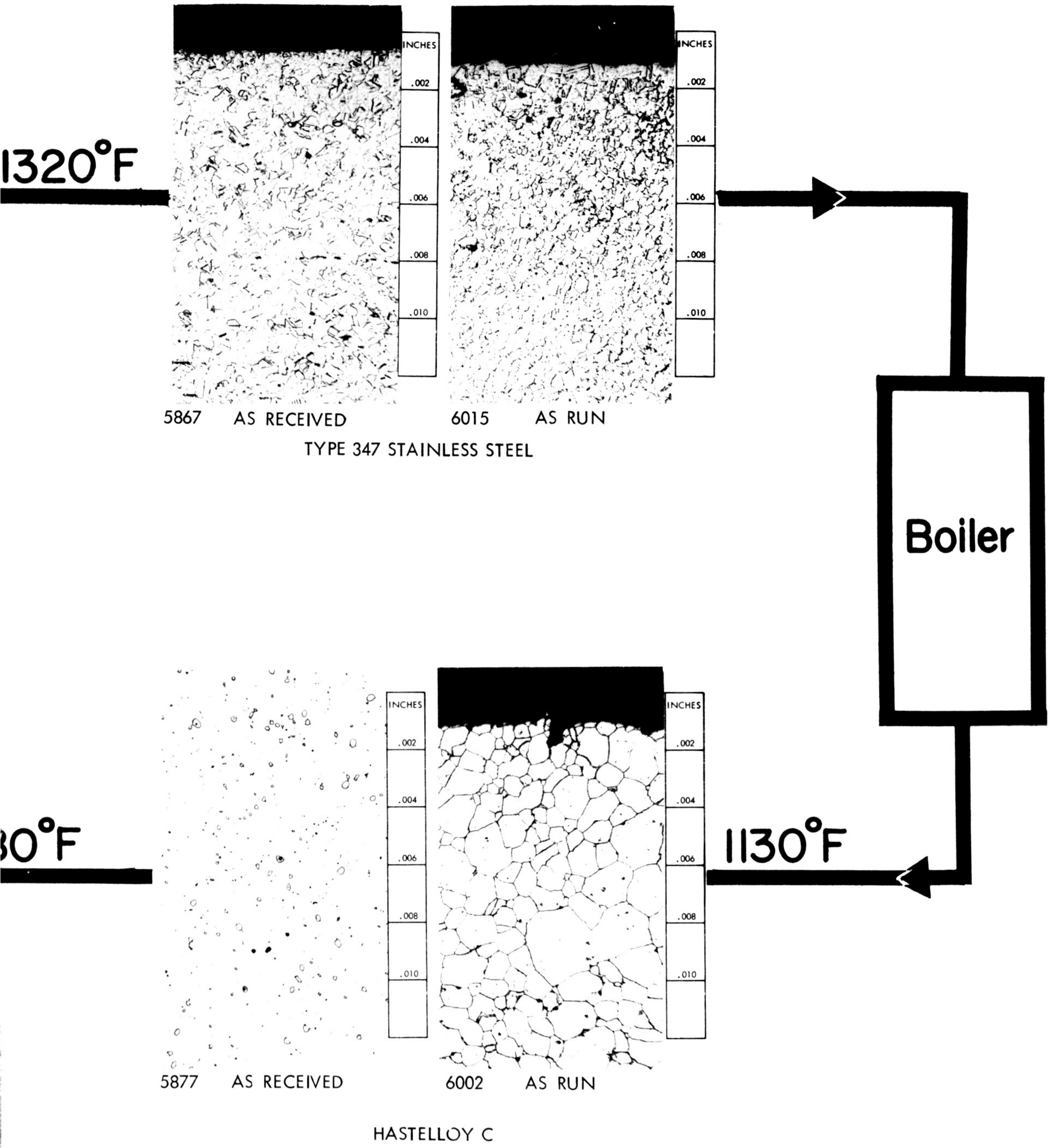

Microstructure of 316 SS Outer Sh

Figure 16



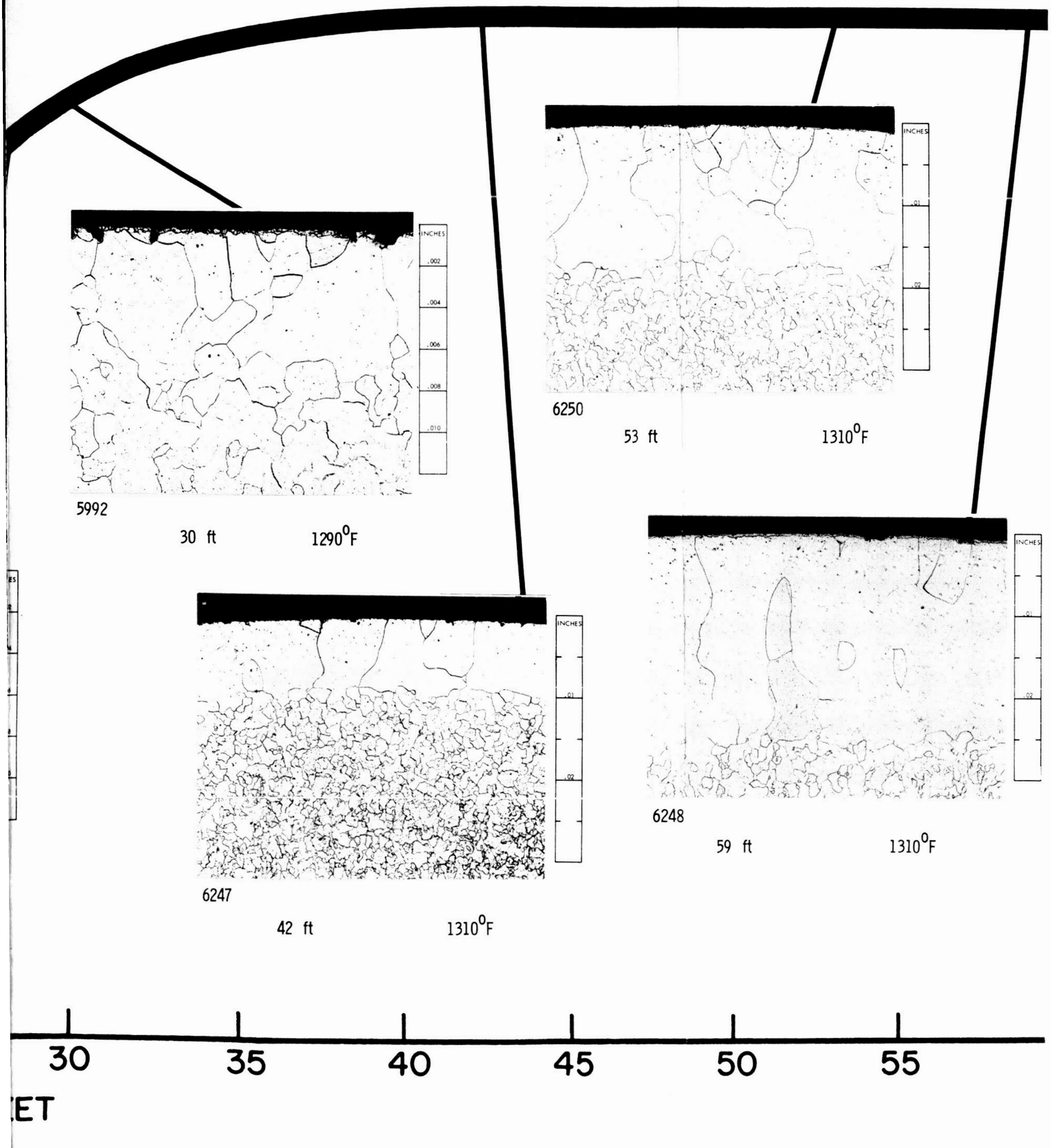

170

Microstructure at Exterior of 9Cr-1Mo S



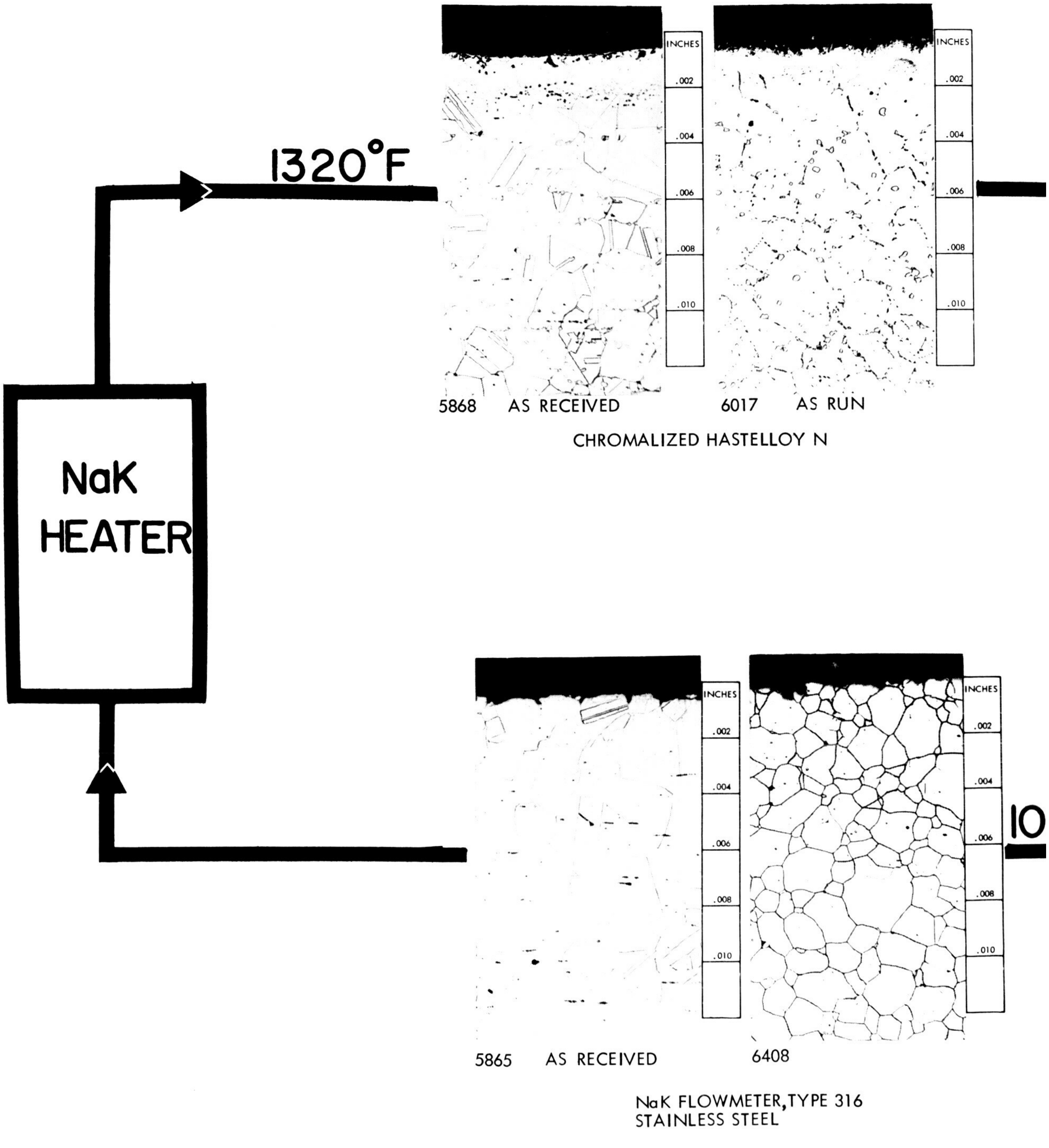

rimary Loop, CL-3 (Vilella's Etch)

Figure 18

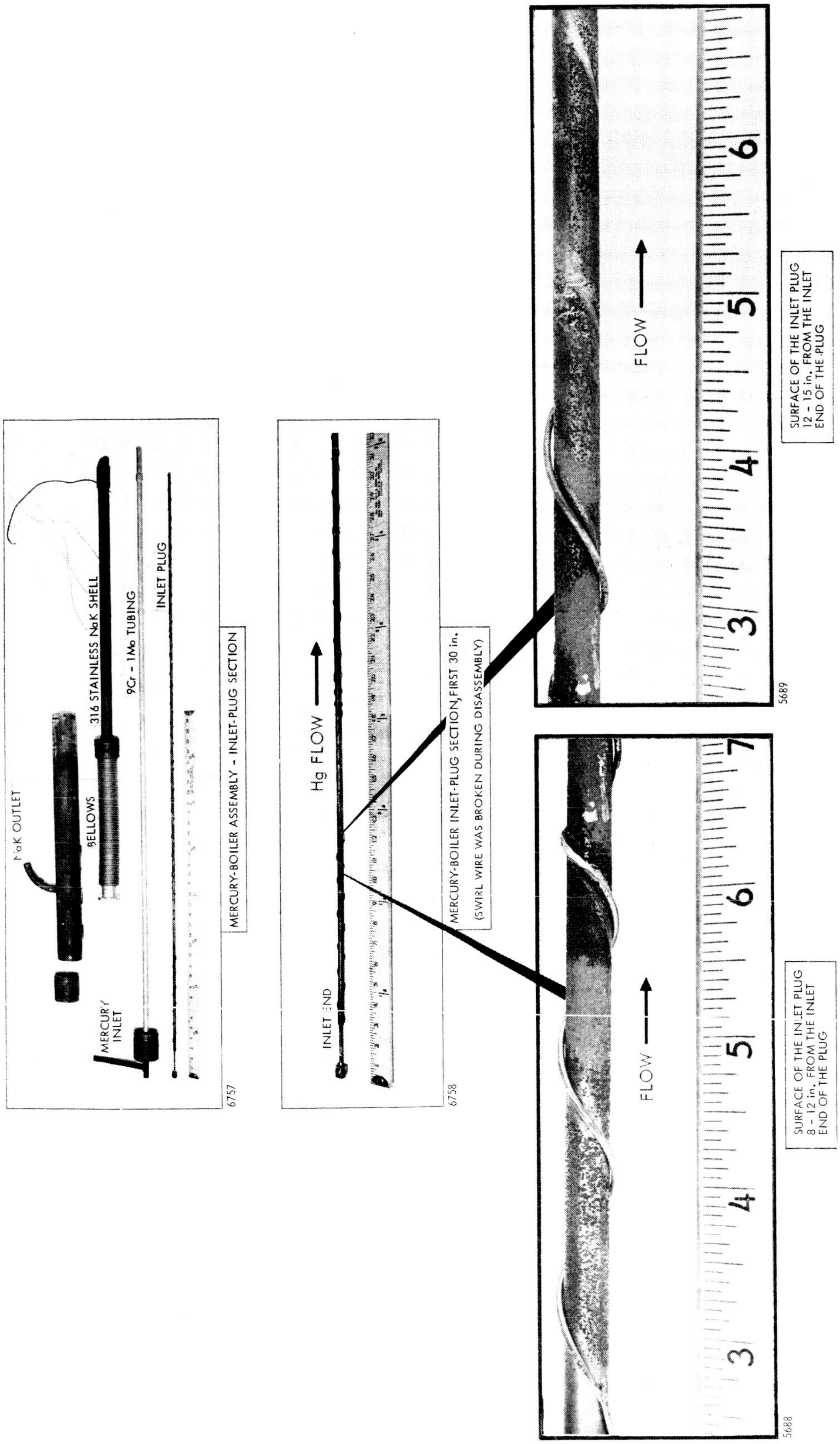



teel Tubing, CL-3 Boiler (Vilella's Etch)

Figure 17

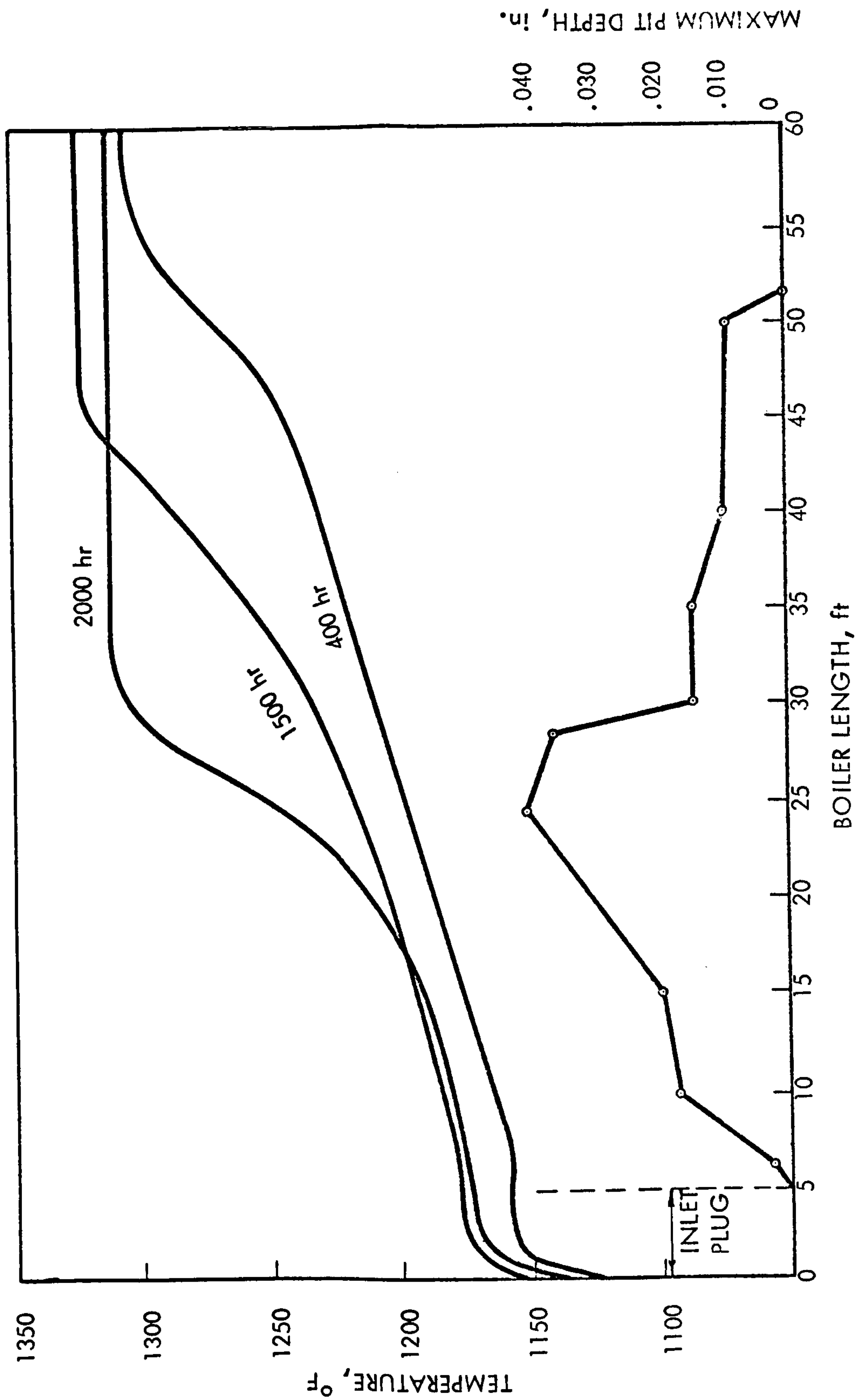



Microstructure of Materials in NaK



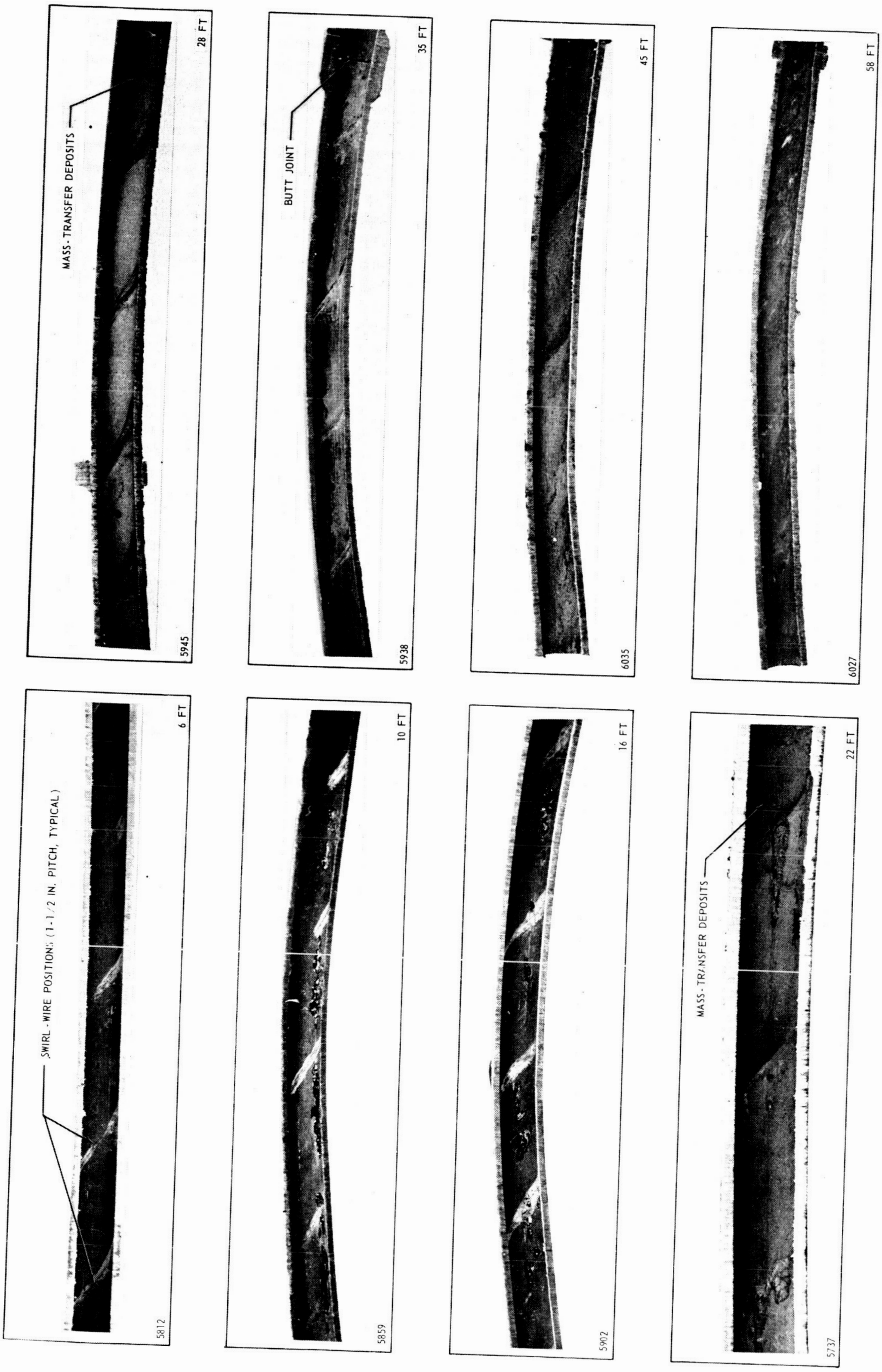

Inlet Plug from CL-3 Boiler

Figure 19




Pitting Depths in 9Cr-1Mo Steel Tubing, CL-3 Boiler

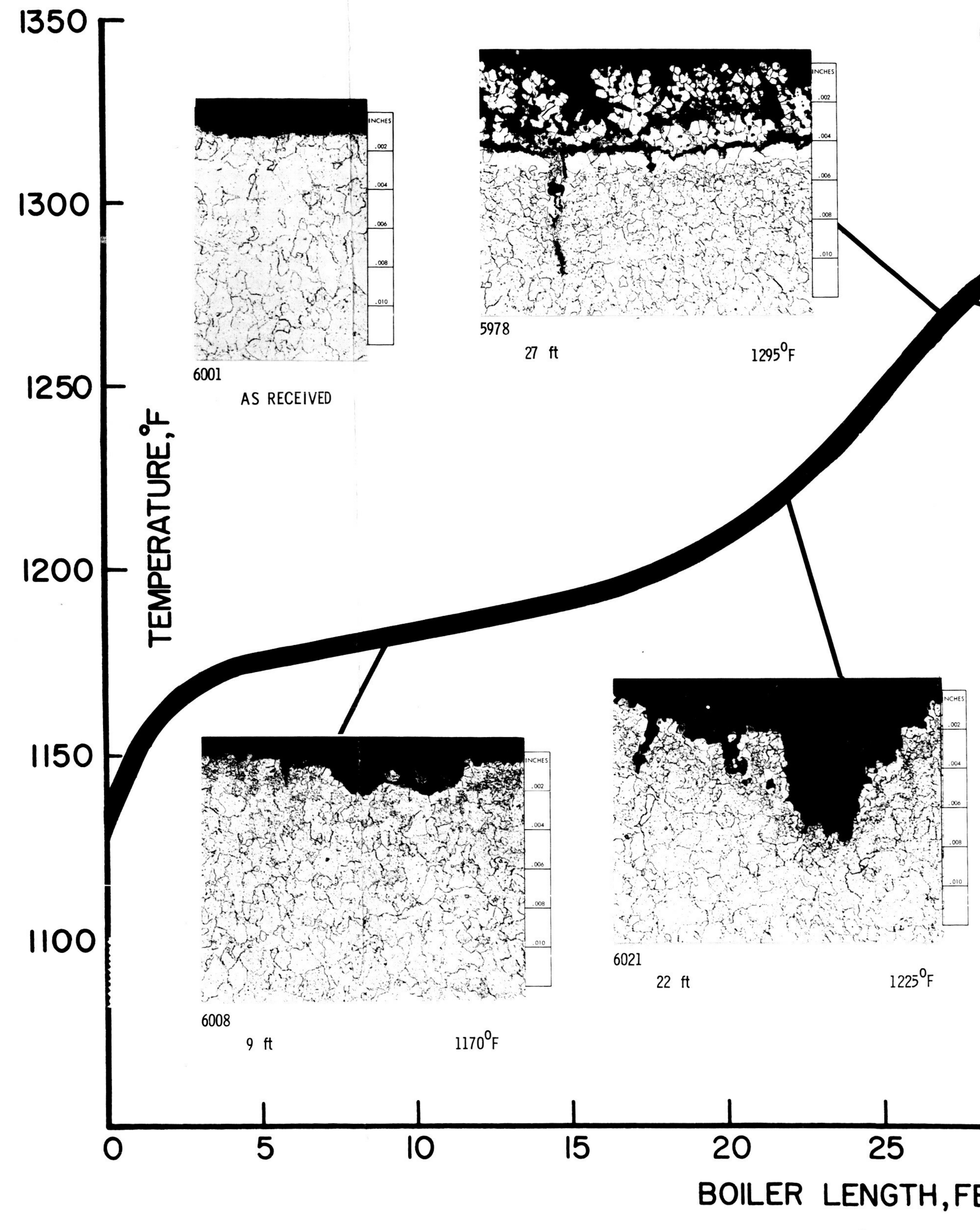
Figure 20




ALL VIEWS: THE MERCURY FLOW IS FROM LEFT TO RIGHT. THE INDICATED LOCATIONS ARE AS MEASURED FROM THE MERCURY INLET.

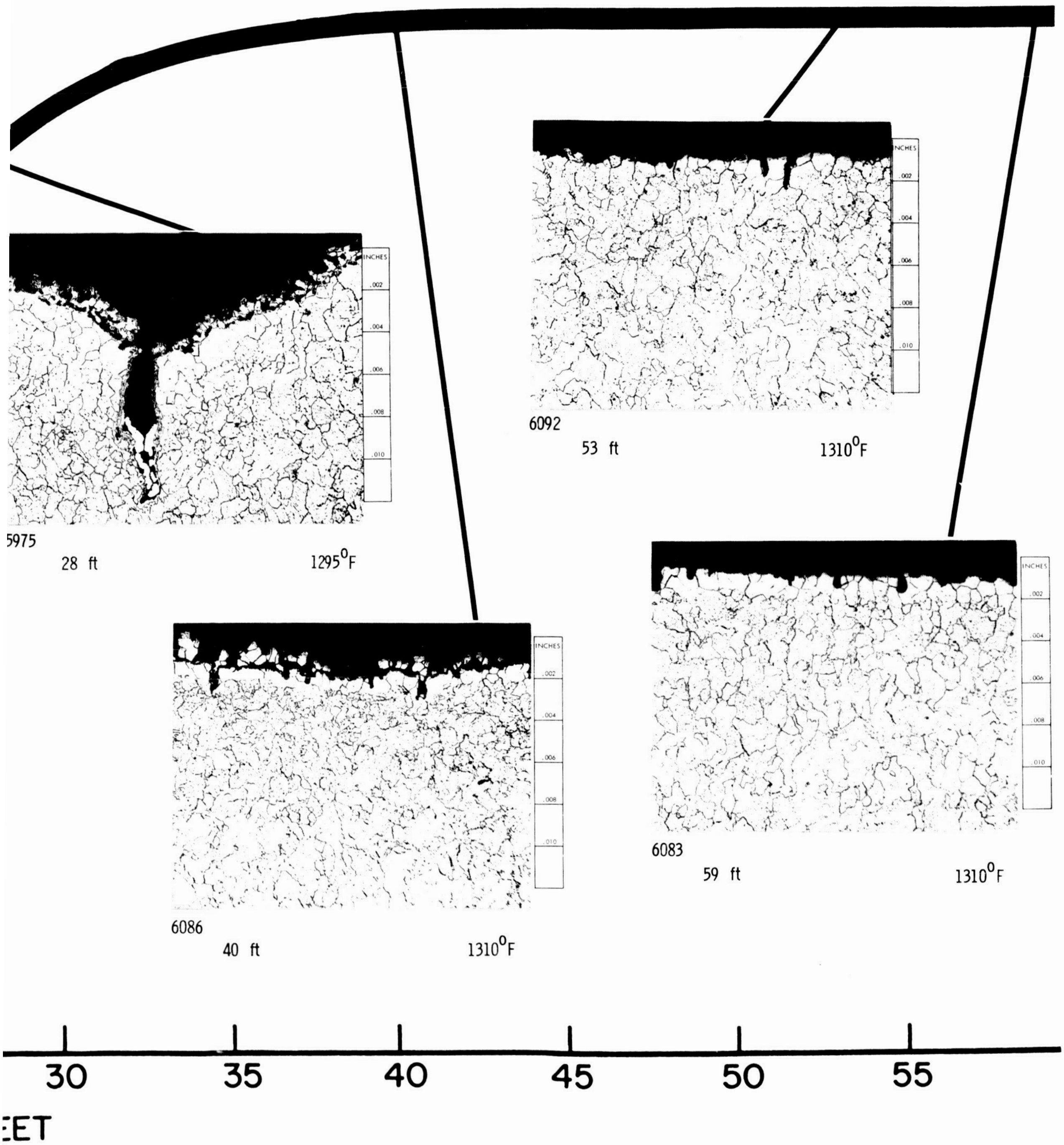
Views of Pitting in 9Cr-1Mo Steel Tubing, CL-3 Boiler

Figure 21

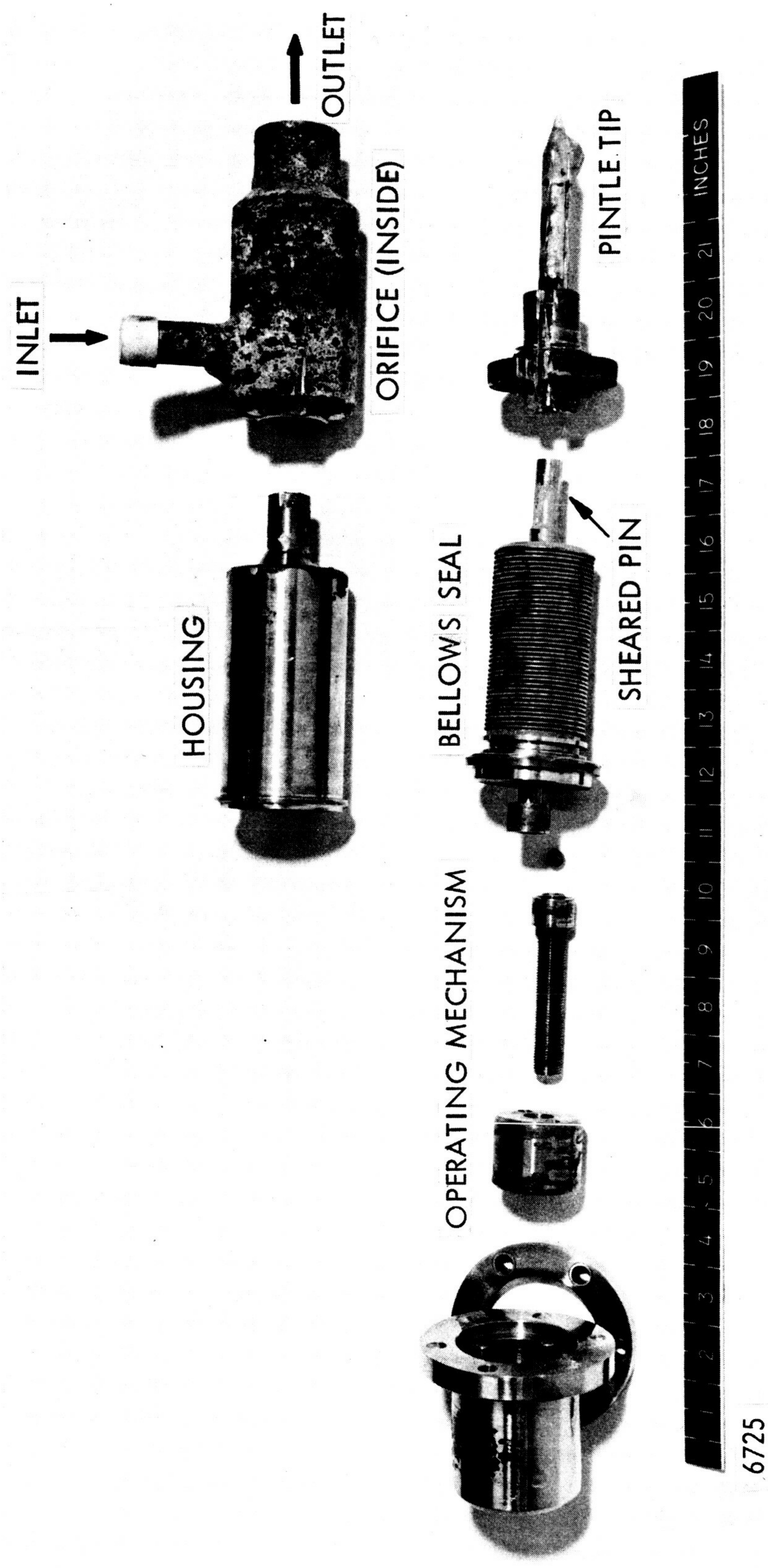



Microstructure at Interior of 9Cr-1Mo St

Figure 22

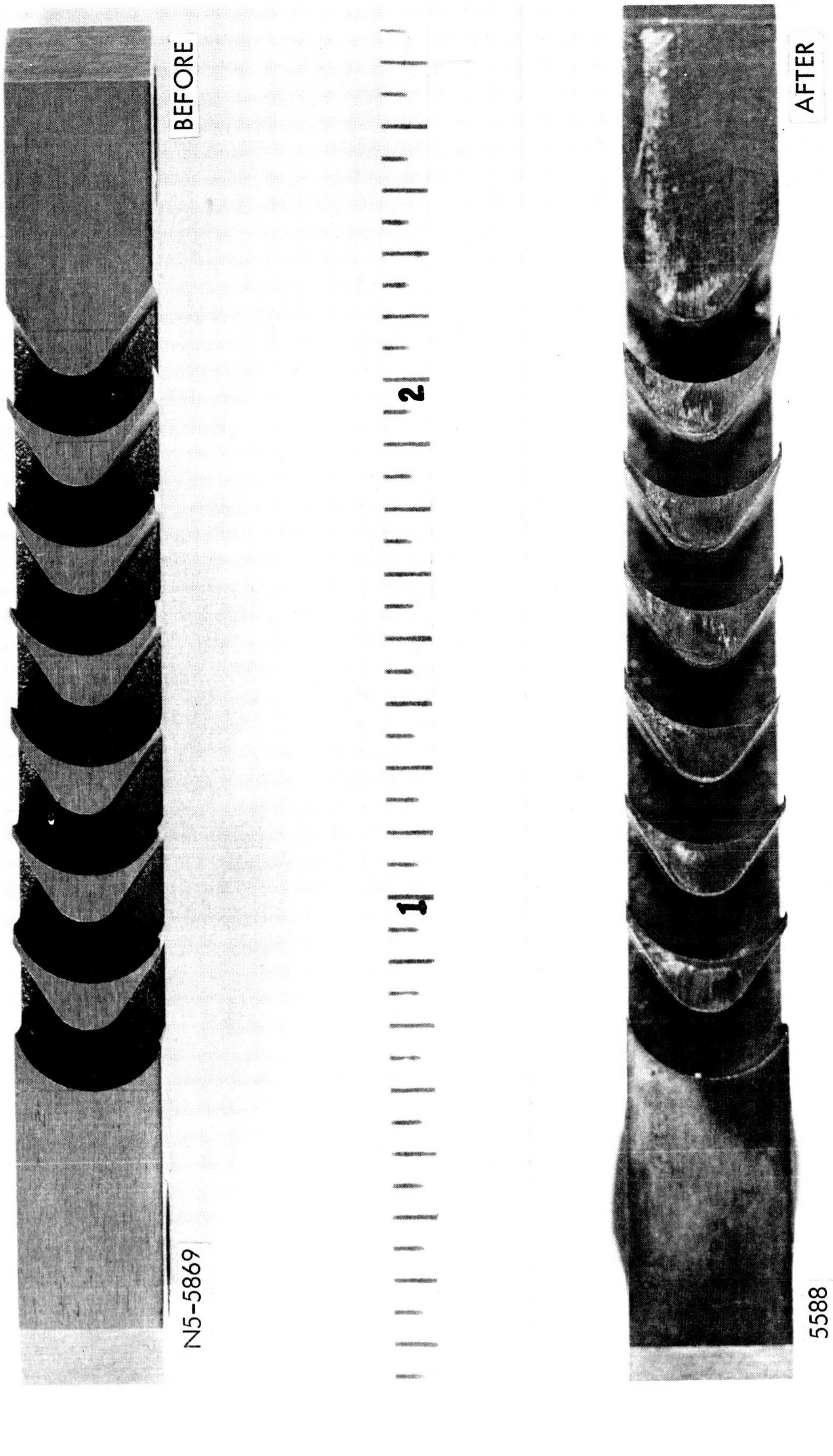



;eel Tubing, CL-3 Boiler (Vilella's Etch)



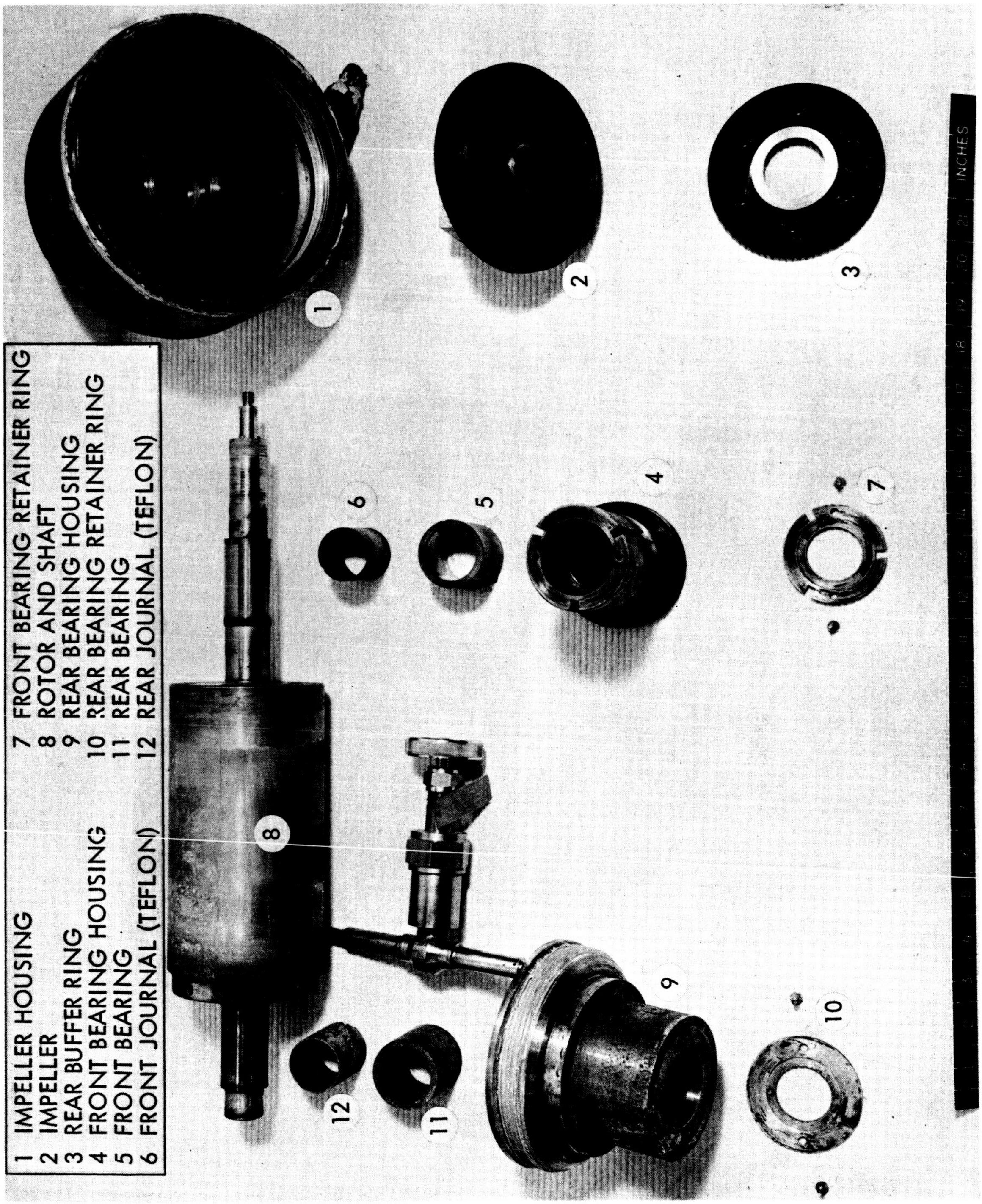

Adjustable Choked Nozzle, CL-3

Figure 23



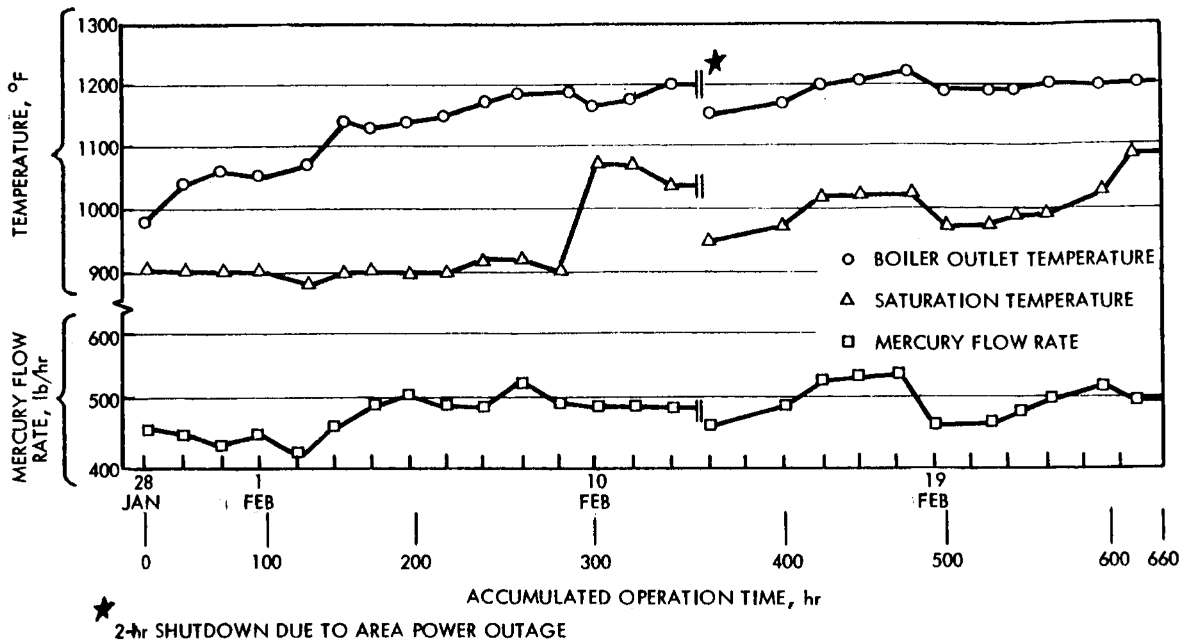

Blade Section Before and After Exposure, CL-3

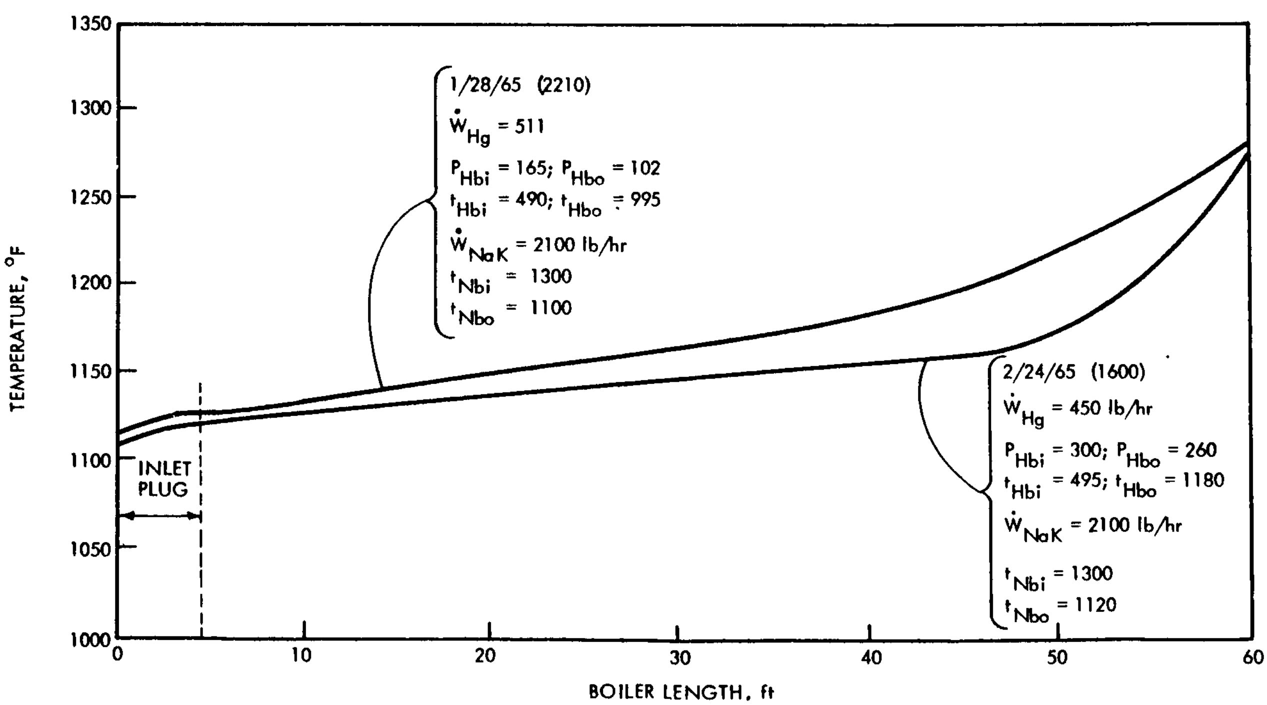
Figure 24



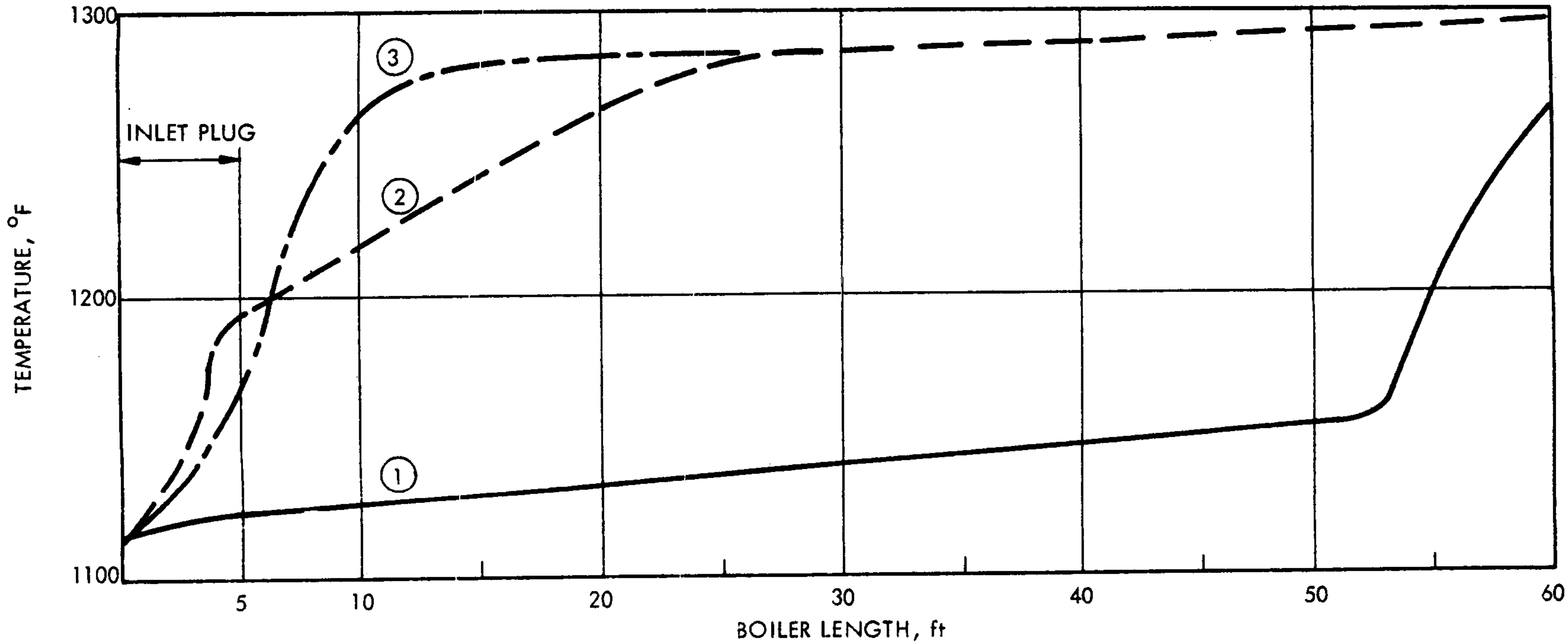

6770

Internal Parts of Mercury Pump (9Cr-1Mo Steel), CL-3




Boiler Performance, CL-4



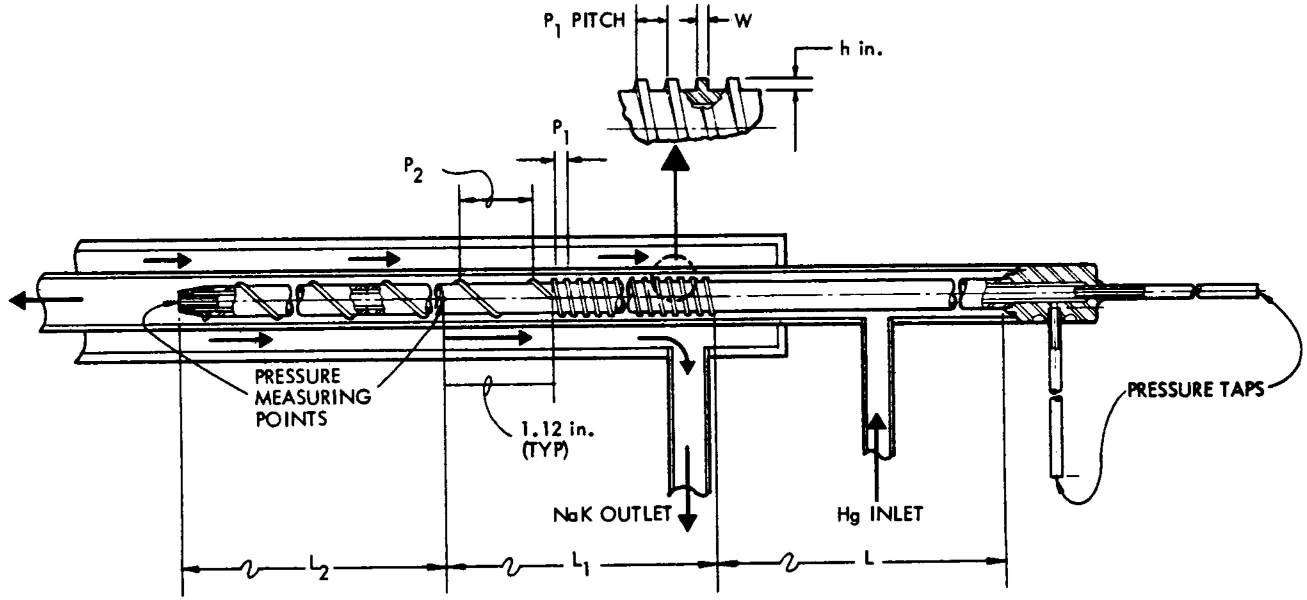

| CURVE | PLUG DISCRIPTION | DATE | NaK $\omega$ lb/hr | NaK $t_{in}$ °F | NaK $t_{out}$ °F | Hg $\omega$ lb/hr | Hg $t_{in}$ °F | Hg $t_{out}$ °F | Hg $P_{in}$ psia | Hg $P_{out}$ psia |
|---|---|---|---|---|---|---|---|---|---|---|
| 1 | 3/4 in. PITCH 0.049 in. WIRE 5 ft LONG | 2/24/65 | 2100 | 1280 | 1120 | 476 | 500 | 1180 | 300 | 260 |
| 2 | MODIFIED PLUG | 3/10/65 | 2100 | 1300 | 1100 | 527 | 390 | 1220 | 340 | 105 |
| 3 | MODIFIED PLUG | 3/17/65 | 2070 | 1305 | 1120 | 500 | 375 | 1225 | 369 | 225 |

NaK-Temperature Profiles, CL-4 Boiler

| PLUG NO. | L | PREHEAT REGION | | | | | LOW QUALITY REGION | | | REMARKS |
|---|---|---|---|---|---|---|---|---|---|---|
| | | TYPE ① | $L_1$ | $P_1$ | W | h | TYPE ① | $L_2$ | $P_2$ | |
| 1 | | W | 60 in. | 0.75 in. | | | | | | NOT INSTRUMENTED |
| 2 | 6 in. | W | 18 in. | 0.125 in. | | | W | 36 in. | 3/4 in. | NOT INSTRUMENTED |
| 3 | 9 in. | M | 15 in. | 0.125 in. | 0.038 | 6.048 | W | 36 in. | 3/4 in. | INSTRUMENTED |
| 3a | 9 in. | W | 15 in. | 0.125 in. | | | W | 36 in. | 1 1/2 in. | INSTRUMENTED |
| 3b | 6 in. | W | 15 in. | 0.17 in. | | | W | 24 in. | 1 1/2 in. | INSTRUMENTED |
| 4 | 9 in. | M | 15 in. | 0.125 in. | 0.038 | 0.045 | W | 36 in. | 1 1/2 in. | INSTRUMENTED |

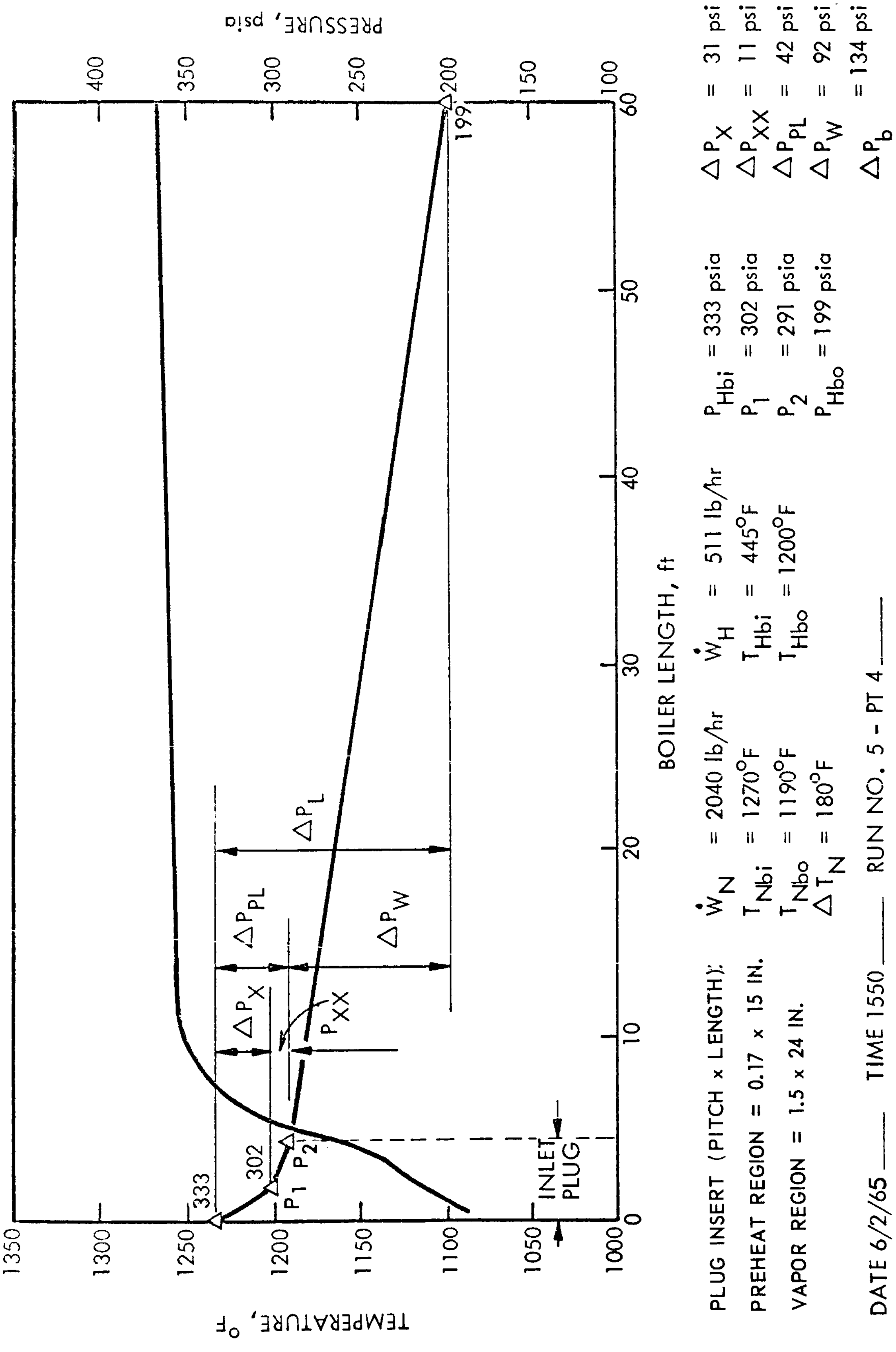
① M = MACHINED THREAD
W = WIRE WOUND

NOTES: a. ALL WIRE USED = 0.049 in. DIAMETER
b. O.D. OF PLUGS = 0.0398 in.




Boiler-Inlet-Plug Configurations

Figure 28




Typical Data Point, Inlet-Plug Test Program

Figure 29

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